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ELEMENTS
OF
CHEMICAL ANALYSIS,
INORGANIC AND ORGANIC.

BY
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TO

THOMAS GRAHAM, ESQ., F.R.S. L. & ED.,

PROFESSOR OF CHEMISTRY IN UNIVERSITY COLLEGE, LONDON,
PRESIDENT OF THE CHEMICAL SOCIETY, ETC.

HAD I no other inducement, my dear Sir, in offering you the present Work, than your high and well-deserved scientific reputation, ample, indeed, would that have been; but the personal attention and kindness you have always manifested towards me constitute not a slight additional incentive to the very small tribute of esteem, the paying of which is now the privilege of

Your late pupil,

and attached friend,

THE AUTHOR.

P R E F A C E.

IN the first chapter and division of this work, instructions are offered for performing the ordinary manipulations of analytical processes; to which are added a description of the most important reagents employed, and the means of detecting the impurities with which these are occasionally contaminated.

The second chapter is devoted to the statement of the appearances produced by the principal reagents when applied to various chemical substances, the results being thrown into a series of tables, and is followed by a chapter containing full instructions for the performance of the qualitative analysis of every variety of substance. This last chapter includes the use in analysis of the mouth blowpipe.

In the fourth and last chapter and division, quantitative analysis is treated of. The processes are there described by which all bodies which enter into the composition of familiar substances are separated and their weights determined; with an account, in conclusion, of the operations of organic analysis, including the recent improvements on the mode of determining the amount of nitrogen.

A large proportion of the processes described are those recommended by our two greatest authorities

in analytical chemistry — Berzelius and Rose, to whose works, especially to the last German edition (1838) of the *Handbuch der Analytischen Chemie* of the latter, the reader is referred for much information which could not be included in the more limited plan of the present work. But several of the processes described are of very recent invention, and new to treatises on the subject. Some of these will be found of considerable value.

An Appendix is added, in which the mode of calculating the atomic constitution of a substance from its per-centage composition, when known, is described, with the methods of taking the specific gravity of matter in different conditions. Numerical tables are also appended, which have been formed to expedite the calculations of analysis.

University College,
July, 1842.

CONTENTS.

CHAPTER I.

MANIPULATIONS AND REAGENTS.

	Page
SECTION I. Manipulations in qualitative Analysis	1
SECTION II. Manipulations in quantitative Analysis	4
SECTION III. Reagents	12
Abbreviations and Symbols	16

CHAPTER II.

BEHAVIOUR OF SUBSTANCES WITH REAGENTS.

Division of metallic Oxides and Acids into Three Classes according to their Behaviour with Sulphuretted Hydrogen and Hydrosulphate of Ammonia	17
Behaviour of Salts of First Class of Oxides with Reagents.	
No. 1.	18, 19
Behaviour of Salts of First Class of Oxides with Reagents.	
No. 2.	20, 21
Behaviour of Salts of Second Class of Oxides with Reagents. A. (Alumina to Oxide of Manganese.) No. 1.	22, 23
Behaviour of Salts of Second Class of Oxides with Reagents. A. No. 2.	24, 25
Behaviour of Salts of Second Class of Oxides with Reagents. B. (Oxide of Manganese to Oxide of Zinc.)	
No. 1.	26, 27
Behaviour of Salts of Second Class of Oxides with Reagents. B. No. 2.	28, 29
Behaviour of Salts of Third Class of Oxides with Reagents. A. (Oxide of Antimony to Iridic Oxide.)	
No. 1.	30, 31
Behaviour of Salts of Third Class of Oxides with Reagents. A. No. 2.	32, 33
Behaviour of Salts of Third Class of Oxides with Reagents. B. (Iridic Oxide to Palladious Oxide.) No. 1.	34, 35
Behaviour of Salts of Third Class of Oxides with Reagents. B. No. 2.	36, 37

	Page
Behaviour of Salts of Third Class of Oxides with Reagents. C. (Palladious Oxide to Peroxide of Tin.)	
No. 1. - - - - -	38, 39
Behaviour of Salts of Third Class of Oxides with Reagents. C. No. 2. - - - - -	40, 41
Behaviour of Third Class of Oxides with Reagents. Acids. D. (Antimonic Acid to Vanadic Acid.) No. 1.	42, 43
Behaviour of Third Class of Oxides with Reagents. Acids. D. No. 2. - - - - -	44, 45
Behaviour of Acids with Reagents. A. (Acetic to Formic.)	46, 47
Behaviour of Acids with Reagents. B. (Formic to Hydrosulphuric.) - - - - -	48, 49
Behaviour of Acids with Reagents. C. (Hydrosulphuric to Perchloric.) - - - - -	50, 51
Behaviour of Acids with Reagents. D. (Perchloric to Tartaric.) - - - - -	52, 53

CHAPTER III.

QUALITATIVE ANALYSIS.

Preliminary Observations. List of Bases and Acids to be sought - - - - -	54
SECTION I. Qualitative Analysis of a Salt consisting of a single Acid and a single Base contained in the List, pages 57. and 58. - - - - -	58
Examination for Base - - - - -	60
Examination for Acid - - - - -	63
SECTION II. Qualitative Analysis of a Compound which can contain all the Bases and Acids in the List, pages 57. and 5. - - - - -	65
Determination of Bases - - - - -	66
I. Examination of the Precipitate by Sulphuretted Hydrogen - - - - -	66
II. Treatment of the Solution filtered from the Precipitate by Sulphuretted Hydrogen - - - - -	67
Treatment of the Solution in Hydrochloric Acid of the Precipitate by Hydrosulphate of Ammonia - - - - -	68
III. Examination of the Solution filtered from the Precipitate by Hydrosulphate of Ammonia - - - - -	69
Determination of Acids - - - - -	70
SECTION III. Qualitative Analysis of a Substance which may contain all inorganic Bodies whose Properties are well known - - - - -	71
I. Examination for Bases and some Acids - - - - -	71
Treatment of the Precipitate formed by Sulphuretted Hydrogen - - - - -	72
Treatment of the Solution filtered from the Precipitate by Sulphuretted Hydrogen - - - - -	73
II. Examination for Acids - - - - -	74

	Page
SECTION IV. Examples of the qualitative Analysis of Metallic	
Alloys - - - - -	75
Analysis of Berlin Silver - - - - -	76
Analysis of Newton's fusible Metal - - - - -	77
SECTION V. Qualitative Analysis of Silicates - - - - -	78
Means of effecting Solution - - - - -	78
Constituents of Silicates - - - - -	80
Analysis of a Silicate insoluble in Hydrochloric Acid, sus- pected to contain an Alkali - - - - -	83
Examples of the qualitative Analysis of Silicates, Stilbite, Hornblende, Lepidolite - - - - -	84
SECTION VI. Qualitative Analysis of Mineral Waters: 1. For the Gases evolved. 2. For the Bodies rendered insoluble by Evaporation. 3. For the Bases in Solution filtered from the Precipitate produced by Ebullition. 4. For the Acids - - - - -	86
SECTION VII. Use of the Blowpipe in qualitative Analysis - - - - -	93
Construction of the Blowpipe, Blowpipe Flame, Blowpipe Supports - - - - -	94
Behaviour of Alkalies and Earths before the Blowpipe	98, 99
Behaviour of Oxides of Metals proper before the Blowpipe (Oxide of Antimony to Oxide of Manganese) - - - - -	100, 101
Behaviour of Oxides of Metals proper before the Blowpipe (Oxide of Manganese to Oxide of Zinc) - - - - -	102, 103
Blowpipe Tests for Acids - - - - -	104
Blowpipe Operations - - - - -	106
First Operation—Heating in a glass Tube closed at one end	106
Second Operation—Heating alone in the open Air - - - - -	107
Third Operation—Heating in a glass Tube open at both ends	108
Fourth Operation—Heating with Reagents: 1. With Car- bonate of Soda. 2. With Borax. 3. With Microcosmic Salt - - - - -	109
SECTION VIII. Analysis of Gases - - - - -	111
Properties of Gases - - - - -	111
Qualitative Analysis of a mixed Gas - - - - -	111
Analysis of Coal Gas (qualitative and quantitative) - - - - -	114
SECTION IX. Detection of Poisons in organic Mixtures - - - - -	115
Arsenic - - - - -	115
Mercury - - - - -	120
Antimony - - - - -	120
Copper - - - - -	120
Lead - - - - -	121
Nitric Acid - - - - -	122
Sulphuric Acid - - - - -	122
Hydrochloric Acid - - - - -	123
Oxalic Acid - - - - -	123
Hydrocyanic Acid - - - - -	124

	Page
SECTION X. Qualitative Analysis of Urine and Discrimination of	
Urinary Calculi - - - -	124
Analysis of Urine - - - -	124
Discrimination (by chemical tests) of Urinary Calculi, consist-	
ing of a single Deposit or of alternating Calculi -	126

CHAPTER IV.

QUANTITATIVE ANALYSIS.

Preliminary Observations - - -	128
SECTION I. Alkalies and their metallic Bases - - -	130
1. Potassium - - - -	130
2. Sodium - - - -	131
Alkalimetry - - - -	132
3. Ammonia - - - -	135
4. Lithium - - - -	135
SECTION II. Alkaline Earths and Earths proper, and their metallic	
Bases - - - -	136
1. Barium - - - -	136
2. Strontium - - - -	137
3. Calcium - - - -	138
4. Magnesium - - - -	139
5. Aluminum - - - -	144
6. Yttrium - - - -	147
7. Thorium - - - -	147
8. Glucinum - - - -	147
9. Zirconium - - - -	149
SECTION III. Metals proper, not precipitated by Sulphuretted	
Hydrogen from their Acid Solutions - - -	149
1. Cerium - - - -	149
2. Manganese - - - -	150
Valuation of Peroxide of Manganese - - -	152
3. Iron - - - -	153
Analysis of Clay Ironstone - - -	155
4. Nickel - - - -	156
5. Cobalt - - - -	159
6. Zinc - - - -	165
7. Uranium - - - -	169
8. Chromium - - - -	170
9. Titanium - - - -	171
SECTION IV. Metals proper, precipitated by Sulphuretted Hydrogen	
from their Acid Solutions - - -	172
1. Lead - - - -	172
2. Cadmium - - - -	174
3. Bismuth - - - -	174

	Page
4. Copper - - - - -	176
5. Silver - - - - -	179
6. Gold - - - - -	180
7. Mercury - - - - -	182
8. Platinum, Palladium, Osmium, Iridium, and Rhodium -	186
9. Tin - - - - -	195
10. Antimony - - - - -	196
11. Molybdenum - - - - -	200
12. Tungsten - - - - -	201
13. Arsenic - - - - -	202
SECTION V. Non-metallic Bodies - - - - -	207
1. Silicon - - - - -	207
Analysis of natural Silicates - - - - -	207
2. Sulphur - - - - -	212
Analysis of Sulphurets - - - - -	214
3. Chlorine - - - - -	217
Chlorimetry - - - - -	218
4. Iodine - - - - -	220
5. Bromine - - - - -	222
6. Fluorine - - - - -	223
7. Phosphorus - - - - -	224
8. Boron - - - - -	227
9. Nitrogen - - - - -	228
Eudiometry - - - - -	230
10. Carbon - - - - -	233
Analysis of Gunpowder - - - - -	235
11. Water - - - - -	237
Analysis of Mineral Waters : Saline, Chalybeate, and Car-	
bonated - - - - -	239
Alkaline Waters - - - - -	247
Sulphureous Waters - - - - -	248
Determination of the Degree of Hardness of Waters -	249
SECTION VI. Organic Analysis - - - - -	254
Determination of Nitrogen - - - - -	264

APPENDIX.

Method of calculating the Atomic Constitution of a Body	
from its per-centage Composition - - - - -	269
Methods of taking the specific Gravity of Bodies - - - - -	270
Addition on Chlorimetry - - - - -	279
Tables for Calculations in Analysis - - - - -	282
Tables of Atomic Weights, with their Logarithms - - - - -	297
General Index - - - - -	301

ELEMENTS OF CHEMICAL ANALYSIS.

CHAPTER I.

MANIPULATIONS AND REAGENTS.

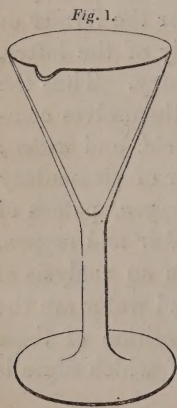
SECTION I.

MANIPULATIONS IN QUALITATIVE ANALYSIS.

THE composition of any substance may be expressed in the simple elements which form its ultimate constituents, and also very frequently by other and less complex bodies, which, by their immediate union, compose the substance in question, and are its proximate constituents. A chemical analysis may have for its object the separation of either the former or latter class of constituents, but most generally of the latter, at least in the department of inorganic chemistry. Thus the substance *alum* is composed of four bodies, themselves compound, namely, *alumina*, *potash*, *sulphuric acid*, and *water*; and each of these is composed again of a pair of elementary bodies, namely, alumina of *aluminum* and *oxygen*, potash of *potassium* and *oxygen*, sulphuric acid of *sulphur* and *oxygen*, and water of *hydrogen* and *oxygen*. Now, in an analysis of alum, the alumina, potash, sulphuric acid, and water are the substances sought, and the elementary composition of these bodies being known, the simple elements of which alum is composed are also determined.

An analysis of any substance may be limited to the discovery of the number and nature of its constituents, or the analysis may extend to the determination of the proportion by weight of these constituents. Thus it might be ascertained that alum contains sulphuric acid, potash, alumina, and water, or that the salt in question consists, in 100 parts, of 33.78 parts of sulphuric acid, 9.93 of potash, 10.82 of alumina, and 45.47 of water. An examination of the first kind, which does not develop more than the nature or quality of the constituents, is termed a *qualitative analysis*; while an examination of the second kind, which embraces the quantities of the constituents, is termed a *quantitative analysis*. The principal manipulations of the two methods will be shortly described separately; the qualitative process first, as it is a necessary introduction to the quantitative process.

The operations to be performed in qualitative analysis are exceedingly simple, and easy of execution, consisting of little more than merely mixing a reagent with the solution of the substance to be analyzed. The *reagent* is so named from its producing a particular appearance or change, the *reaction*, by which the presence of a particular substance is indicated. The reagent is also often referred to as the *test* or *test-liquid*. In all ordinary cases, the reagent is applied in solution to the substance to be analyzed, the latter being also dissolved in its proper solvent. The best vessels to per-

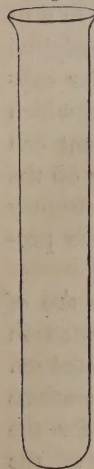


form the mixture in, are test glasses, resembling ordinary conical wine glasses, provided with a spout for the convenience of pouring liquids into a tube or flask, with the view of applying heat, an operation frequently necessary. The liquids are mixed in the test glasses by stirring them together briskly with a glass rod. A test glass should be clear and colourless; it should neither run down to a point within, nor should the bottom be flat, but gently rounded. Such a glass is represented in *fig. 1*. If the quantity of material to be

operated on be small, the mixture with the reagent may be made on a flat piece of glass, using only one or two drops of the solution of the substance to be analyzed, and applying the test from the extremity of a glass rod. The same thing may be done in a watch-glass, where it is necessary to apply heat; or if the reagent produce a change of colour, in a small porcelain capsule, held in any convenient manner over a gas or a spirit-lamp flame.

It is advisable, however, in general, to operate on small quantities, and to apply the test drop by drop, as it frequently happens that a very different effect is produced by the same reagent when added in large, to what is produced in small quantities; differences which are highly characteristic, and only perceived when the test is applied in a gradual manner. Instead of test glasses, test tubes (*fig. 2.*) are frequently employed, possessing as they do the advantage over glasses of allowing heat to be applied without transferring the liquid into

Fig. 2.



another vessel. A convenient size for ordinary use, is five inches in length, by three quarters of an inch in diameter. The mixture of the two substances must be effected by agitation.

The effects exhibited by some particular tests are not immediately perceptible, but are seen only after a certain time has elapsed: in such a case the mixture containing the reagent must be allowed to stand a few minutes, a mark distinguishing what test has been added, being applied to prevent mistakes.

It is often necessary in qualitative analysis to ascertain the action of a test liquid on a *precipitate* which has been produced in the course of testing: this may, in general, be done in the same test glass or tube in which the first testing was performed, the supernatant liquid, if the precipitate subside readily, being poured off. Stirring with a glass rod, and application of heat, frequently assist the subsidence of a precipitate. When the supernatant liquid contains something in solution which interferes with the action of the

liquid to be added, the precipitate must be filtered and washed with distilled water, until perfectly cleaned from the interfering substance. This can be performed by means of a small paper filter (page 6.) held in a glass funnel, some appropriate test being applied to ascertain when the liquid which passes through the filter is perfectly pure.

SECTION II.

MANIPULATIONS IN QUANTITATIVE ANALYSIS.

The manipulations necessary in quantitative analysis are, in general, equally simple, but must be performed with extreme care and attention. The ingredient whose amount is to be ascertained is commonly either (1.) *separated from its solution in the solid form*, or (2.) it is *expelled from a known weight of the material in a state of purity*. In the first instance, it is collected with several precautions, and obtained in a fit state for weighing; in the second, the weight of the original substance being known, the weight of the particular substance can be ascertained from the loss of weight which occurs on its expulsion. The amount of one ingredient can also be estimated indirectly when the whole quantity of the other ingredients has been obtained, the loss or deficiency on the original weight then evidently representing the proportion present of that particular substance.

By far the larger number of analytical operations are of the first description; that is, a substance is separated in the solid state from a solution of the material operated on. General directions for the performance of such operations only will be given at present; the manipulations for the others will be described afterwards, when treating of the particular substances to which such operations refer.

Precipitation from a solution is the means usually employed to obtain an ingredient of a compound in a solid or fixed form. The original combinations of the material to be analyzed, or of the substances into which it may be con-

verted by the act of solution, are then broken up : each particular ingredient whose amount is the object of inquiry being either (1.) combined with another substance, and forming a new insoluble compound (in which it is contained in known proportions, and which is capable of being weighed), or (2.) being in its own natural condition insoluble, although held in solution by the substance to which it was united in the material under examination, that combination is destroyed, the ingredient assumes its natural insoluble condition, and is therefore precipitated. Thus in the analysis of chloride of mercury (corrosive sublimate), for instance, to obtain the *chlorine*, the salt named is dissolved in water, and nitrate of silver added to the solution. The combination of mercury with chlorine is thus destroyed, not a particle of chloride of mercury remaining, if sufficient nitrate of silver has been added. The chlorine unites with silver, forming a new insoluble compound, chloride of silver, capable of being weighed, and containing constant and known proportions of chlorine and silver. Next, to obtain the *mercury*, to a solution of the chloride of mercury, protochloride of tin is added. The original combination of mercury with chlorine is, as before, broken up ; the protochloride of tin acquiring all the chlorine of the chloride of mercury, that metal is set free, precipitates, and may be collected and weighed. From the weight then of the chloride of silver (100 parts of which contain 24.67 parts of chlorine), the amount of chlorine becomes known : the mercury is ascertained by direct weighing.

The first point to be attended to in precipitation is, that both the solution of the substance to be analyzed, and of the precipitant or reagent employed, be made perfectly clear by filtration ; for it is obvious that any solid matter diffused through either of the solutions would be carried down by, and afterwards separated with, the precipitate whose weight is to be ascertained, thus producing a fallacious result.

To add precisely the proper quantity of the precipitant is difficult, but rarely necessary ; an excess in most cases producing no greater inconvenience than a little longer washing on the filter ; to be assured that a sufficiency has

been added, the precipitate must be allowed to subside, and the reagent dropped carefully into the supernatant liquid, when it will be seen whether an additional precipitate is formed or not. If a precipitate is formed, of course a further quantity of the precipitant must be added, and the examination repeated.

The subsidence and aggregation of a precipitate are favoured in various ways: the most usual is by heating the liquid; so advantageous, indeed, is this means, that it would be advisable to follow as a general rule, always to precipitate while warm, and to boil the liquid gently for a few minutes after precipitation. Next to heating, agitation with a glass rod, and the addition of an acid or an alkali, are the means usually adopted; but the employment of the latter must be guided by the nature of the particular substances operated on.

Fig. 3.



glass beaker vessel, that is, a jar thin at bottom and with a

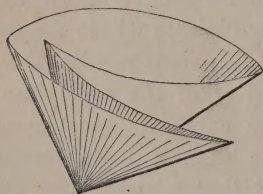
Fig. 4.



When heat is to be applied, a convenient vessel to perform the precipitation in, is a porcelain bason, not very shallow, having a capacity of about twelve or fourteen ounces (*fig. 3.*). A spreading edge at top (*fig. 4.*), is also very useful for applying heat; but where heat is not requisite, a common glass tumbler, having a spout for convenience in pouring from, may be employed. It will be found useful to have different sizes of these tumblers and

beakers. It is unnecessary for the bottoms of the tumblers to be more than one eighth of an inch thick.

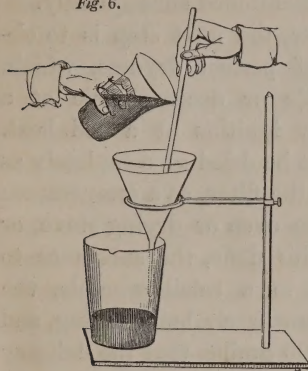
Fig. 5.



The next operation in order is *filtration*. This is done by means of a paper filter, formed by doubling twice a circular piece of filtering paper so as to form a quadrant, and then opening one of the folds, as shown in *fig. 5.* The filter

is supported in a glass funnel, which is itself held in any convenient manner, a vessel being placed below the funnel to receive the filtered liquid.* Before pouring the turbid

Fig. 6.



liquid on it, the filter should be wetted with a few drops of distilled water, which has the effect of swelling the fibres of the paper, without which precaution the liquid would not at first pass through clear. As the loss of a single drop of the liquid might render the operation worthless, its transference to a filter must always be directed by a glass rod applied to the lip of the containing vessel, and

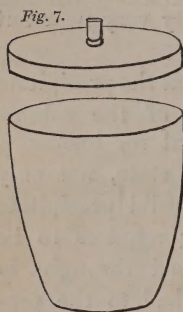
held nearly perpendicular, with its extremity very near but not touching the filter (*fig. 6.*).

It invariably happens in precipitations, that the precipitate carries down with it a sensible quantity of the soluble salts present in the solution (the precipitant for instance), so that to obtain the precipitate in a pure state, not only must the liquid itself be washed from the solid precipitate with pure distilled water, but the washing requires to be continued long after the solution has passed through, in order to remove all the soluble salt attached to the precipitate. This is done most effectually by hot water, an appropriate reagent being occasionally applied to the washings to ascertain whether the salts are still being washed out. A delicate indication of the presence of any salt which is not volatile, is afforded by evaporating a few drops to dryness on a clean piece of platinum foil, a minute trace of the salt causing a very perceptible stain, which could not occur with pure distilled water. So long as this stain is observed, the

* An excellent filtering paper is sold by Messrs. R. Griffin and Co. of Glasgow, from whom filters of various sizes, already cut, may also be procured. They likewise supply all the other articles required in analysis.

washing must be continued. There are a few precipitates which, being slightly soluble, must not be washed for a continued length of time: the cases in which this precaution is necessary to be observed will be mentioned subsequently.

Supposing the washing complete, the next step is to obtain the precipitate in a state of perfect dryness, which, if the substance be not volatile or decomposable at a high temperature, is effected by ignition at a red heat. Before ignition, however, it must be dried as completely as possible, without removing from the filter, at a temperature of about 180° or 200° Fahr., in an oven or drying stove, or in a basin on the sand-bath. This done, the substance to be weighed must be transferred to a small crucible, the weight of which is ascertained immediately before using, and which should not exceed 300 or 400 grains, that the delicacy of the balance may not be impaired. For ordinary use, a

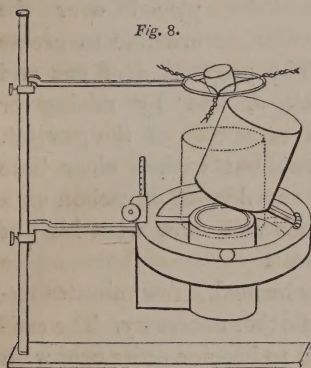


small platinum crucible (*fig. 7.*) is by far the most convenient, a common spirit lamp with a large flame affording in general a sufficient degree of heat. When a substance is to be ignited which might corrode platinum, a crucible of Berlin porcelain is employed. The transference of the precipitate to the crucible, without losing the minutest portion, is the most delicate part of the whole operation. Place the crucible,

with its lid removed, in the centre of a sheet of highly glazed coloured paper; with the assistance of a clean platinum spatula transfer the bulk of the precipitate to the crucible, and hold the filter by means of a forceps at that part which, being folded, contains none of the precipitate, immediately over the crucible. Apply a light to the filter, and burn it completely, allowing the precipitate to fall into the crucible, and so regulating the combustion that the flame shall reach last that part of the filter held by the forceps, otherwise (unless the crucible be large enough to retain it) it will fall on the glazed paper while burning. In this way, it sometimes happens that the filter may be completely burned,

although more frequently a carbonaceous residue remains, which must be afterwards burned by allowing air to have access, by lifting the cover when the crucible has obtained a full red heat.

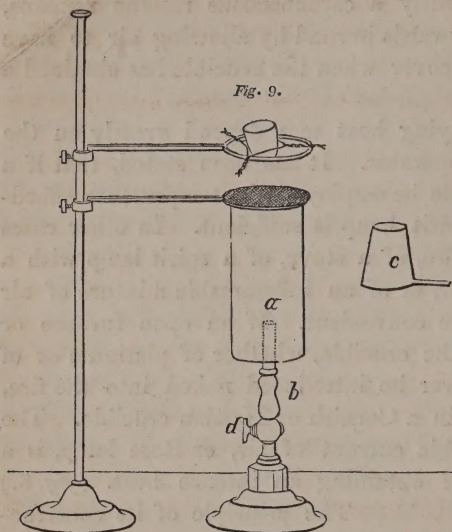
The mode of applying heat may depend greatly on the convenience of the operator. It has been stated, that if a small platinum crucible be employed, the temperature afforded by an ordinary spirit lamp is sufficient. In other cases the heat of an open fire, of a stove, of a spirit lamp with a double current of air, or of an inflammable mixture of air and coal gas, is more convenient. If an open furnace or stove be employed, the crucible, whether of platinum or of porcelain, should never be introduced naked into the fire, but always enclosed in a Cornish or Hessian crucible. The spirit lamp with double current of air, or Rose lamp, is a very ready means of obtaining an intense heat. (*fig. 8.*)



The principle of its construction is the same as that of the Argand oil lamp, a current of air being supplied to the interior as well as to the exterior of the flame. In the figure, the chimney is represented attached to the lamp by a hinge, by which means ready access to the wick is obtained for the purpose of trimming. The only objection to the use of

this lamp is the large quantity of spirit it consumes.

Where coal gas can be commanded, an excellent source of heat is the combustion of an inflammable mixture of gas and air, which affords a degree of heat equal to that produced by the Rose lamp, at a much less expense. The mixture of gas and air is made in the chimney; a wire gauze is fastened over the top, above which the inflammable mixture is ignited. The annexed cut (*fig. 9.*) represents the entire arrangement: *a* is a copper chimney supported by a retort ring, having a ridge or pegs for this purpose on the outside:



the bottom is open to admit air; *b* is the gas jet, the extremity of which is near the bottom of the chimney. The crucible to be ignited is supported over the flame on a triangular piece of iron wire placed on another ring of the retort stand; *c* is a second chimney or jacket, to be placed over the crucible to create

a constant and equal draught. The proportion of gas and air is regulated by the stopcock *d*, and by raising or depressing the chimney over the extremity of the gas jet. The flame should not burn yellow, but with a clear blue colour, and ought not to produce any deposit of carbon on a body held in it. A little experience will show what kind of flame produces the most intense heat.

If the filter has been completely burned, a few minutes exposure to a red heat is all that is farther necessary. The crucible may then be removed, allowed to become quite cold with its cover still on, and weighed. The weight of the empty crucible must be ascertained, both immediately before and after the ignition: if any appreciable difference exist in the two weighings, and its cause is not obvious, the analysis must be rejected.

The ash of the filter in which the precipitate was collected has been weighed with the latter, for which an allowance must obviously be made. If the weight of the ash was not previously known, a similar filter to the one used may be burned, and the ash weighed by itself, or placed with the weights in the oppo-

site pan of the balance. The most convenient way is to use filters of particular sizes and weights, and to have previously ascertained the proportion of ash contained in the paper by burning a known weight, calcining the residue in a platinum capsule, and weighing.

Such is the course to be adopted in the drying and ignition of precipitates which are not volatile at a red heat. When, on the contrary, the precipitates are volatile, as for instance, calomel and sulphuret of mercury, or when they undergo partial decomposition at a high temperature, a different routine must be followed. The filter in which the substance is collected must then be weighed before filtering, and the substance and filter weighed together after drying; but some precautions are required to do this with accuracy. Before a filter is weighed, it must be rendered perfectly dry by holding it for a few minutes near the fire; it is then introduced folded, either into a platinum crucible carefully closed with its lid, or into a glass tube sealed at one end, and closed at the other with a cork, the outside of which is coated with sealing-wax. This being weighed, the filtration may be proceeded with. The precipitate and filter, washed, and dried in an oven at a temperature of about 200° , or in a water-bath at 212° , is introduced into the same crucible or tube, and weighed. After a second heating, if the weight be the same as at first, the substance may be considered as dry; if not, the heating must be continued until the weight becomes constant. The increase on the original weight of the filter and containing vessel gives, of course, that of the substance.

Such are the general directions which can be given for the performance of the ordinary operations of precipitation, filtration, and ignition. But there are many analytical operations which must be performed in a manner entirely different from what has been described; and others, in which a modification merely of the process is necessary. These, however, are so numerous and varied, that a description of the manipulations of each method would constitute almost an entire description of the processes, which must necessarily be

reserved for that portion of the work which treats of actual analysis. We proceed, then, to the consideration of the characters of chemical substances by which they are distinguished from each other, or to the subject of qualitative analysis.

This discrimination, as has been stated, is effected by the application of certain tests or reagents, every substance which can be presented for examination behaving in a peculiar manner with some one or other test; and there are few bodies which present precisely the same appearance with any single test. These characters are expressed in the tables which immediately follow; before studying which, however, it will be necessary to mention the principal reagents employed, their ordinary impurities, and the means of detecting them.

SECTION III.

REAGENTS.

Sulphuric acid. — A few drops evaporated on a platinum capsule should not leave a fixed residue. Oil of vitriol ought not to become turbid on diluting it with four or five times its bulk of water. If it does, this arises from sulphate of lead, which is soluble in strong, but not in the dilute acid. By dilution and subsidence, the sulphate of lead can be removed, and the acid used as a test. If wanted pure in a concentrated state, it must be obtained by evaporating the acid which has been diluted and decanted from the precipitated sulphate of lead, or by distilling oil of vitriol.

Solution of *sulphate of soda* can be used for most of the purposes to which sulphuric acid is applied as a test.

Hydrochloric acid should be colourless [*iron*, and *organic matters**], and should not produce a precipitate with salts of barytes [*sulphuric acid*]. Neutralized with ammonia, it ought not to become black when hydrosulphate of ammonia is added [*iron*]. It may be obtained pure by diluting com-

* The name of the impurity adverted to is placed in brackets.

mercial muriatic acid until of the specific gravity 1.11, and distilling. Of this density it distils without gaining or losing strength.

Nitric acid should not leave a fixed residue on evaporation: it should not give precipitates with chloride of barium [*sulphuric acid*], nor with nitrate of silver [*chlorine*]. If it contain these impurities, add a little nitrate of silver, and distil.

Oxalic acid is impure if it deliquesces or leaves a fixed residue on ignition. Its crystals should be colourless, and their solution should not give a precipitate with salts of barytes. This acid is easily purified by repeated crystallizations. *Binoxalate of potash* and *oxalate of ammonia* are also employed. The former is sometimes adulterated with bitartrate of potash and with bisulphate of potash; the first impurity is detected by heating the salt with oil of vitriol; if it become dark-coloured, and evolve sulphurous acid, it contains either bitartrate of potash or some organic impurity. Bisulphate of potash is detected by a solution of a salt of barytes. Oxalate of ammonia should not leave any incombustible residue.

Tartaric acid. — The usual impurities of tartaric acid are sulphuric acid and a salt of lime. A moderately dilute solution should not produce in salts of barytes a precipitate insoluble in nitric acid; nor should oxalate of ammonia cause a precipitate after the acid has been neutralized by ammonia. Tartaric acid must be purified by re-crystallization.

Potash. — Solution of caustic potash is usually contaminated with carbonate of potash, sulphate of potash, and chloride of potassium. If it contain carbonate of potash, it effervesces when saturated with an acid. After being neutralized with nitric acid, it ought not to give a precipitate with nitrate of silver [*chlorine*], nor with nitrate of barytes [*sulphuric acid*]. By evaporation until of the spec. grav. 1.250, all the sulphate of potash is deposited: the supernatant liquid, decanted, may be diluted for general use; but it will then probably contain some carbonate. If required absolutely pure, it must be made from carbonate of potash prepared from the bicarbonate or the bitartrate of potash.

Ammonia frequently contains muriate, carbonate, and sulphate of ammonia, chloride of calcium, and, very rarely, a trace of oxide of tin. The solution should be limpid and colourless [*organic matters*]; neutralized with nitric acid, it should neither be precipitated by nitrate of silver nor by chloride of barium, and should leave no fixed residue on evaporation.

Carbonate and bicarbonate of potash.—The impurities are, alkaline sulphates and chlorides, silica and alumina. The tests of purity are, after being neutralized with nitric acid, nitrate of silver [*chlorine*], and a soluble salt of barytes [*sulphuric acid*]. If the neutralized solution, when evaporated to dryness, and the residue treated with water, leave white flocks or a gelatinous substance undissolved, this is silica. If alumina be present, the neutralized solution will give a white precipitate with carbonate of ammonia.

Bicarbonate of potash should not precipitate sulphate of magnesia in the cold.

Carbonate of soda.—Besides sulphates and chlorides, this may contain an alkaline sulphuret, sulphite, or hyposulphite. When neutralized by an acid, the gas evolved should be quite inodorous [*sulphuretted hydrogen*, and *sulphurous acid*], and no white precipitate of sulphur should be produced.

Carbonate of ammonia.—Impurities and means of detection are the same as those for ammonia.

Nitrate of silver.—Its ordinary impurities are a salt of copper and nitrate of potash. The former is detected by the solution assuming a blue colour when an excess of ammonia is added. The whole of the silver being precipitated by hydrochloric acid, the filtered liquid should not afford a fixed residue on evaporation [*nitrate of potash*].

Chloride of barium and nitrate of barytes.—Their impurities are, chlorides of calcium and of strontium, nitrate of strontian and iron. The dry salts, moistened with alcohol, should not burn with a red flame; they should not deliquesce in the air, nor produce a blue precipitate with yellow prussiate of potash.

Sulphuretted hydrogen may be said to be the most impor-

tant of all reagents. For purposes of qualitative analysis, it may be kept in solution in water, the solution being made with distilled water, which has been recently boiled to expel air (as the oxygen of the air decomposes sulphuretted hydrogen when in solution), and preserved in carefully closed bottles. In the absence of sulphuretted hydrogen water, and when a small quantity only of the reagent is required, it is conveniently applied by generating the gas in a test tube, moistening a piece of paper with the solution to be tested, and holding the latter within the tube. Sulphuret of iron, if pure, is more convenient for making this gas than sulphuret of antimony, as the former does not require the application of heat.

Hydrosulphate of ammonia is easily prepared by transmitting sulphuretted hydrogen gas through solution of ammonia until no more of the former is absorbed. It should be preserved in well closed bottles which do not contain lead.

Muriate of ammonia. — Its ordinary impurities are sulphate of soda, sulphate of ammonia, and chloride of sodium. It should sublime without residue, and a salt of barytes should produce no precipitate in its solution.

Acetate of lead. — Its impurities are acetate of lime and acetate of iron. The whole of the lead being separated from its solution by sulphuretted hydrogen, no precipitate should be produced in the filtered solution by ammonia, hydrosulphate of ammonia [*iron*], nor by oxalic acid [*lime*].

Protochloride of tin. — Its ordinary impurities are salts of iron and of lead. Hydrosulphate of ammonia should entirely redissolve the precipitate it occasions in the solution of protochloride of tin. If a black insoluble residue remain, this is either sulphuret of iron or sulphuret of lead.

Chromate of potash occasionally contains some sulphate of potash. This is detected by the precipitate produced in the solution by nitrate of barytes not being entirely soluble in free nitric acid. Chromate of barytes is soluble, but sulphate of barytes is insoluble in nitric acid.

Acetate of barytes. — Its ordinary impurity is chloride of barium. This is detected by nitrate of silver, which, if

moderately dilute, affords no precipitate with pure acetate of barytes.

Iodide of potassium. — Its common impurities are carbonate of potash and alkaline chlorides. Its solution should not effervesce with acids. To detect any chloride, the following method may be followed: — Precipitate the solution of the iodide completely by nitrate of silver, add excess of ammonia, and filter. If any chloride of silver was formed, this is now in the filtered liquid, held in solution by ammonia, and may be precipitated by nitric acid; but as iodide of silver is not perfectly insoluble in ammonia, a faint turbidity on adding nitric acid to the filtered ammoniacal solution, may always be expected.

ABBREVIATIONS AND SYMBOLS

EMPLOYED IN THE FOLLOWING TABLES.

am.	-	-	-	-	ammonia.
bicarb.	-	-	-	-	bicarbonate.
carb.	-	-	-	-	carbonate.
CO ₂	-	-	-	-	carbonic acid.
conctd.	-	-	-	-	concentrated.
cryst.	-	-	-	-	crystalline.
ex.	-	-	-	-	excess.
gelat.	-	-	-	-	gelatinous.
HCl.	-	-	-	-	hydrochloric acid.
insol.	-	-	-	-	insoluble.
mur. am.	-	-	-	-	muriate of ammonia.
NO ₅	-	-	-	-	nitric acid.
O	-	-	-	-	no precipitate.
pot.	-	-	-	-	potash.
ppt.	-	-	-	-	precipitate.
ppn.	-	-	-	-	precipitation.
SO ₃	-	-	-	-	sulphuric acid.
sol.	-	-	-	-	soluble.
soln.	-	-	-	-	solution.
solns.	-	-	-	-	solutions.
vol.	-	-	-	-	voluminous.

CHAPTER II.

BEHAVIOUR OF SUBSTANCES WITH REAGENTS.

Division of Metallic Oxides and Acids into Three Classes, according to their Behaviour with Sulphuretted Hydrogen and Hydrosulphate of Ammonia.

I. METALLIC oxides not precipitated by sulphuretted hydrogen, nor by hydrosulphate of ammonia:—

Potash	Lithia	Strontian	Magnesia
Soda	Ammonia	Barytes	Lime.

II. Precipitated by hydrosulphate of ammonia, but not by sulphuretted hydrogen (as sulphurets) in an acid solution:—

Alumina	Lanthanum, oxide of	Uranium, protoxide of
Cerium, oxide of	Manganese, deutoxide of	Uranium, peroxide of
Chromium, oxide of	Manganese, protoxide of	Vanadium, oxide of
Cobalt, oxide of	Nickel, oxide of	Ytria
Glucina	Tantallic acid	Zinc, oxide of
Iron, peroxide of	Titanic acid	Zirconia.
Iron, protoxide of	Thorina	

III. Precipitated by sulphuretted hydrogen from acid solutions:—

a. Oxides having basic properties:

Antimony, oxide of	Lead, oxide of	Platinic oxide
Bismuth, oxide of	Mercury, protoxide of	Platinous oxide
Cadmium, oxide of	Mercury, suboxide of	Rhodium oxide
Copper, protoxide of	Molybdous oxide	Silver, oxide of
Copper, suboxide of	Molybdic oxide	Telluric oxide
Gold, oxide of	Osmic oxide	Tin, peroxide of
Iridic oxide	Palladious oxide	Tin, protoxide of.

b. Oxides having acid properties:

Antimonious acid	Hypermanganic acid (in neutral solutions)*	Osmic acid
Antimonious acid		Selenious acid
Arsenic acid	Manganic acid (neutral)	Tungstic acid
Arsenious acid	Molybdic acid	Vanadic acid.

* Hypermanganic and manganic acids should, strictly, be included in the Second Class; but their characters as *acids* are more conveniently exhibited in the tables by connecting them with the Third Class.

Behaviour of Salts of FIRST CLASS

	Caustic Potash.	Ammonia.	Carbonate of Soda.
Potash.		0	0
Soda.	0	0	0
Lithia.	0	0	In concd. solns. after long standing, a white ppt.
Ammonia.	Evolves am., as- certained by odour and by fumes - with HCl.		0
Barytes.	White, sol. in much water ; becomes tur- bid on expo- sure to the air.	0 The mixture absorbs CO ₂ from the air, and deposits carb. barytes.	White, sol. in acids with ef- fervescence.
Strontian.	As barytes.	As barytes.	As barytes.
Lime.	Ditto.	Ditto.	Ditto.
Magnesia.	White, vol. al- most wholly sol. in mur. am.	White, sol. in mur. am.; 0 if a salt of am. be present, unless boiled.	Bulky white ; sol. in mur. am.

OF OXIDES *with Reagents.* No. 1.

Bicarbonate of Potash.	Carbonate of Ammonia.	Alcoholic Solution of Chloride of Platinum.	Strong Solution of Tartaric Acid.
0	0	Light yellow on stirring. Insol. in acids.	White, cryst. sol. in alkalies, in HCl, SO ₃ , and NO ₅ .
0	0	0	0
0		A faint troubling in strong solns.	0
0	0	Clear yellow ppt. on stirring. Insol. in acids.	If conctd. white cryst. sol. in alkalies.
White, in strong solns. Bicarb. barytes.	White. Carb. barytes.	Yellowish, granular.	White, in strong solns.
As barytes.	As barytes.	0	0
Ditto.	Ditto.	0	0
0 If boiled, a white ppt.	0 If boiled, a white ppt. sol. in mur. am.	0	0

Behaviour of Salts of FIRST CLASS

	Phosphate of Soda.	Hydrofluosilic Acid.
Potash.	No precipitate.	Very gelat. ppt. insol. in HCl.
Soda.	Ditto.	If solns. be very strong, a gelat. ppt. is formed.
Lithia.	Ditto. After adding am. a white ppt. falls on standing.	A white ppt.
Ammonia.	No change.	0, except in very strong solns. Caustic ammonia precipitates silicic acid.
Barytes.	White, sol. in NO_3 and HCl.	On standing gives a cryst. ppt., nearly insol. in HCl. and NO_5 .
Strontian.	As barytes.	No precipitate.
Lime.	Ditto.	Ditto.
Magnesia.	White ppt. ; especially on adding am. or on boiling.	Ditto.

OF OXIDES *with Reagents.* No. 2.

Oxalic Acid.	Sulphuric Acid.	Remarks. — Other Tests.
0	0	Carbazotic acid produces a yellow cryst. ppt. Perchloric acid gives a white ppt., insol. in alcohol.
0	0	Salts of soda communicate a yellow colour to flame; use alcohol or blowpipe flame.
0	0	See Blowpipe Tables (coloured flames).
0	0	All salts of ammonia, except those having fixed acids (phosphate, borate, &c.), volatilize without residue when heated.
0 Oxalate of ammonia gives a white ppt.	White; sulphate of barytes. Insol. in dilute acids.	Sol. of sulphate of lime precipitates solns. of salts of barytes.
White, if conctd. Gives a ppt. with oxalate of ammonia, if dilute.	White; sulphate of strontian. Slightly soluble in water.	Sol. of sulphate of lime gives no ppt. Communicate red colour to the flame of alcohol.
White; increased on adding am. Sol. in NO_5 and HCl.	White, of sulphate of lime in strong solns. Slightly sol. in water.	Communicate red colour to the flame of alcohol.
0 except when solns. be very strong.	0	Characteristic test is phosphate of soda with ammonia.

Behaviour of Salts of SECOND CLASS

	Potash.	Carbonate of Potash.
Alumina.	White, vol., sol. in ex., re-pptd. by mur. am.	White, vol., insol. in ex., insol. in mur. am.
Cerium, oxide of.	White, vol., insol. in ex.	White, vol., slightly sol. in ex.
Chromium, oxide of.	Clear green ppt. sol. in ex. re-pptd. by boiling.	Clear green, becoming blue ; sol. in ex.
Cobalt, oxide of.	Blue, insol. in ex. ; be- comes green and red on exposure.	Red ; becomes blue on boiling.
Glucina.	White, vol. sol. in ex. re-pptd. by mur. am.	Bulky white ; sol. in large ex. insol. in mur. am.
Iron, peroxide of.	Ochre-red, insol. in ex.	Ochre-red.
Iron, protoxide of.	First white, then green, and lastly brown.	Green, becoming red ; sol. in mur. am.
Lanthanum, oxide of.	Bulky white.	Bulky white.
Manganese, prot- oxide of.	White, becoming brown and black.	White, does not blacken unless ignited : slightly sol. in mur. am.
Manganese, deut- oxide of.	Bulky brown.	Bulky brown.

OF OXIDES *with Reagents.* A. No. 1.

Bicarbonate of Potash.	Ammonia.	Carbonate of Ammonia.
Effervescence ; vol. white ; insol. in mur. am.	Vol. white ; insol. in ex. ; insol. in mur. am.	Vol. white, insol. in ex.
White, vol., slightly sol. in ex.	Bulky white, insol. in ex.	Bulky white ; slightly sol. in ex.
Clear green.	Violet.	Clear green.
Red.	First blue ; on adding more am. green : slightly sol. in ex. ; sol. in mur. am.	Red, sol. in mur. am.
Bulky white ; sol. in large ex.	Bulky white ; insol. in ex. ; insol. in mur. am.	Bulky white, sol in ex.
Ochre-red ; effervescence.	Ochre-red ; insol. in ex.	Ochre-red.
Green becoming red ; effervescence.	Green, becoming red ; sol. in mur. am.	Green becoming red ; sol. in mur. am.
Gelat. ; white, no efferv.	Bulky white ; insol. in ex. ; insol. in mur. am.	Bulky white, insol. in ex.
White ; formed slowly if sol. is dilute, or if it contain mur. am.	White ; blackens on exposure ; sol. in mur. am. This soln. deposits peroxide of manganese on standing.	White ; does not blacken ; slightly sol. in mur. am.
Brown.	Deep brown.	Brown.

Behaviour of Salts of SECOND CLASS

	Yellow Prussiate of Potash.	Red Prussiate of Potash.	Hydrosulphate of Ammonia.
Alumina.	0	0	White, sol. in potash. (alumina.)
Cerium, oxide of.	White.	0	White. (oxide.)
Chromium, ox- ide of.	0	0	Greenish. (oxide of chromium.)
Cobalt, oxide of.	Green ; insol. in HCl.	Brownish red ; insol. in HCl.	Black insol. in ex. (sulphuret.)
Glucina.	0	0	White, sol. in potash. (glucina.)
Iron, peroxide of.	Deep blue ; in- sol. in HCl, sol. in alkalies.	0	Black, insol. in ex. ; becomes brown on ex- posure. (sulphuret.)
Iron, protoxide of.	White, becom- ing blue, in- sol. in HCl.	Deep blue, insol. in acids.	Black, insol. in ex. ; becomes brown on ex- posure. (sulphuret.)
Lanthanum. (solution of chloride.)	Dense white, insol. in HCl.	0	White. (oxide.)
Manganese, protoxide of.	White, with a shade of pink ; sol. in acids.	Brown, insol. in acids.	Flesh colour, in- sol. in ex., blackens on exposure. (sulphuret.)
Manganese, deutoxide of.	Greenish.	Brown.	Flesh-colour, in sol. in ex. (sulphuret.)

OF OXIDES *with Reagents.* A. No. 2.

Oxalic Acid.	Phosphate of Soda.	Remarks. — Other Tests.
0	Bulky white ; sol. in potash ; sol. in acids.	Sulphate of potash and a little free sulphuric acid give crystals of alum.
White ; sol. in much HCl.	White.	Organic bodies impede pptn. by alkalies.
0	Green in neutral solutions.	Ditto. (See Blowpipe Tables.)
White ; formed slowly.	Blue in neutral solutions.	Organic bodies impede pptn. by alkalies, but not by hydrosulph. am. (See Blowpipe Tables.)
0	Bulky ppt. in neutral solns.	Salts of glucina have a characteristic sweet taste.
0 Liquid becomes yellow.	White, in neutral solns. ; sol. in am.	Infusion of gall nuts gives a black ppt. Characteristic test is yel. pruss. pot.
Yellow ; — formed slowly ; sol. in HCl.	White.	Inf. of gall nuts gives no black ppt. Characteristic test is red pruss. pot.
White, sol. in HCl.	White, sol. in HCl.	Chloride has an astringent taste.
White, cryst. ; formed slowly ; sol. in SO_3 and HCl.	White. Does not change colour.	Solution of chloride of lime gives a brown ppt. of perox. manganese if no ammoniacal salt is present. (See Blowpipe.)
0	Bulky brown in neutral solns.	

Behaviour of Salts of SECOND CLASS

	Potash.	Carbonate of Potash.
Nickel, oxide of.	Apple-green, insol. in ex.	Apple-green.
<i>Tantalie acid.</i> (Solution in oxalate of potash.)	White.	White.
<i>Titanic acid.</i> (Solution of alkaline titanate in a small quantity of HCl.)	White, sol. in HCl.	White, slightly sol. in ex., sol. in HCl.
Thorina.	Gelatinous white, insol. in ex.	White, sol. in large ex.
Uranium, oxide of.	Bulky brown, insol. in ex.	Greenish, sol. in ex.
Uranium, peroxide of.	Yellow, insol. in ex.	Yellow, sol. in ex. re-pptd. on standing.
Vanadium, oxide of.	Dirty white, sol. in ex. re-pptd. by a greater ex.	Dirty white, sol. in ex. re-pptd. by a greater ex.
Yttria.	Vol. white, insol. in ex.	White, vol. slightly sol. in ex.
Zirconia.	Bulky white, insol. in ex.	Bulky white, slightly sol. in ex.
Zinc, oxide of.	Gelat. white, sol. in ex.	White; insol. in ex.; sol. in potash, am. and mur. am.

OF OXIDES *with Reagents.* B. No. 1.

Bicarbonate of Potash.	Ammonia.	Carbonate of Ammonia.
Apple-green ; effervescence.	Green, sol. in ex.	Apple-green, sol. in ex.
White.	White.	White.
White, slightly sol. in ex., sol. in HCl.	White, insol. in ex., sol. in HCl.	White, slightly sol. in ex., sol. in HCl.
White, sol. in conctd. ex.	Gelat. white, insol. in ex.	White, sol. in. conctd. ex.
Greenish, sol. in ex.	Dark brown, insol. in ex.; becomes yellow on exposure.	Greenish, sol. in ex.
Yellow. insol. in ex.	Yellow.	Yellow, very sol. in ex.
Grey, sol. in ex.	Brown, insol. in ex.	Grey, becoming brown.
Bulky white, sol. in ex.	Bulky white, insol. in ex.	Bulky white, sol. in ex.
Bulky white, slightly sol. in ex.	Bulky white, insol. in ex.	Bulky white, more sol. in ex.
White; effervescence.	Bulky white, sol. in ex.	White, sol. in ex.

Behaviour of Salts of SECOND CLASS

	Yellow Prussiate of Potash.	Red Prussiate of Potash.	Hydrosulphate of Ammonia.
Nickel, oxide of.	Greenish white, insol. in HCl.	Yellowish green, insol. in HCl.	Black, slightly sol. in ex. (sulphuret.)
<i>Tantalie acid.</i> (Soln. in oxalate of potash.)	Yellow.		White, efferves- cence. (tan- talic acid.)
<i>Titanic acid.</i> (Soln. of al- kaline tita- nate in a little HCl.)	Deep green.		White. (titanic acid.)
Thorina.	White, sol. in acids.	0	White. (thorina.)
Uranium, ox- ide of.	Brown red.	Brown red, formed slowly.	Black, insol. in ex. (proto- sulphuret.)
Uranium, per- oxide of.	Brown red.	0	Brown, very slightly sol. in ex. (persul- phuret.)
Vanadium, oxide of.	Yellow, insol. in acids; becomes green.	Gelatinous green.	Brown black, sol. in ex. (sul- phuret.)
Yttria.	White.	0	White. (yttria.)
Zirconia.	White.	0	White, bulky. (zirconia.)
Zinc, oxide of.	Bulky white, insol. in HCl.	Yellowish red, sol. in HCl.	White, insol. in ex. (sulphuret.)

OF OXIDES *with Reagents.* B. N^o. 2.

Oxalic Acid.	Phosphate of Soda.	Remarks. — Other Tests.
Yellowish, formed slowly.	Light green.	Organic bodies impede pptn. by alkalies, but not by hydrosulph. am. (See Blowpipe Tables.)
O		Tincture of gall nuts gives an orange-yellow ppt.
White, sol. in large ex., sol. in HCl.		A rod of zinc, in a soln. of an alkaline titanate in HCl, slowly forms a blue ppt. Tinct. galls gives an orange-yellow ppt.
White, insol. in ex., slightly sol. in acids.	White.	
Yellowish green.	Greenish white.	Organic bodies impede pptn. by alkalies.
	White, sol. in acids.	Org. bodies impede pptn.
		Org. bodies impede pptn. by alkalies, but not by hydrosulph. am.
Bulky white, sol. in HCl.	White, sol. in acids, re-pptd. on boiling.	Org. bodies impede pptn. by alkalies.
Bulky white, sol. in HCl.	Bulky white.	Ditto.
White, sol. in acids and alkalies.	White, sol. in acids, and alkalies.	Hydrosulph. am. is a characteristic test.

Behaviour of Salts of THIRD CLASS

	Potash.	Carbonate of Potash.	Bicarbonate of Potash.
Antimony, oxide of.	White, insol. in ex.	White, insol. in ex.	White, insol. in ex.
Bismuth, oxide of.	White, insol. in ex.	White, insol. in ex.	White, insol. in ex. ; effervescence.
Cadmium, oxide of.	White, insol. in ex.	White, insol. in ex.	White ; effervescence.
Copper, sub-oxide of.	White ; becomes black on exposure.	Yellow.	Yellow.
Copper, protoxide of.	Blue, insol. in ex. ; becomes black on boiling.	Blue ; becomes black on boiling.	Green, sol. in ex., giving blue solution.
Gold, oxide of.	Slight black on standing.	0	0
Iridic oxide.	Solution decolorized ; very slight brown ppt.	Brown red.	Becomes decolorized ; no ppt.

OF OXIDES *with Reagents.* A. No. 1.

Ammonia.	Carbonate of Ammonia.	Yellow Pruss. of Potash.	Red Pruss. of Potash.
White, insol. in ex.	White, insol. in ex.	White, insol. in HCl.	Faint turbidity on standing.
White, insol. in ex.	White, insol. in ex. ; effervescence.	White, insol. in HCl.	Faint yellow, sol. in HCl.
White, very sol. in ex.	White, insol. in ex.	Yellowish white, sol. in HCl.	Yellow, sol. in HCl.
Soln. becomes blue on exposure.	Soln. becomes blue on exposure.	White, becomes brown on exposure.	Red brown.
Blue, very sol. in ex., giving a deep blue solution, re-pptd. by potash.	Green ; sol. in ex., giving blue soln.	Reddish brown, insol. in HCl.	Yellowish green, insol. in HCl.
Yellow. (fulminating gold.)	Yellow ; effervescence. (fulminating gold.)	Soln. becomes emerald green.	0
Soln. decolorized, slight brown ppt.	Soln. decolorized after some time.	Soln. is decolorized.	0

Behaviour of Salts of THIRD CLASS

	Hydrosulphate of Ammonia.	Sulphuretted Hydrogen.	Oxalic Acid.
Antimony, oxide of.	Orange red, sol. in ex.	Orange red in acid solns.	White, sol. in HCl.
Bismuth, oxide of.	Black, insol. in ex.	Black.	White crystals on standing.
Cadmium, oxide of.	Yellow, insol. in ex.	Yellow, insol. in alkalies.	White, very sol. in am.
Copper, suboxide of.	Black, insol. in ex.	Black or brown.	White, becomes blue on exposure.
Copper, protoxide of.	Black.	Black.	Greenish.
Gold, oxide of.	Dark brown, sol. in ex.	Black.	Deep brown.
Iridic oxide.	Brown, sol. in ex.	Brown, formed slowly.	Soln. decolorized after some time.

OF OXIDES *with Reagents.* A. No. 2.

Phosphate of Soda.	Bar of Metallic Zinc.	Iodide of Potassium.	Remarks.
White, sol. in HCl.	Black powder. (metallic anti-mony.)		Chloride of antimony gives a white ppt. of subchloride on pouring into water.
White.	Black powder. (reduced bismuth.)	Brown, sol. in ex.	Unless very acid, solns. of bismuth are decomposed by dilution, subsalts being pptd.
White.	Grey spangles of cadmium.		See Blowpipe Tables.
White, becoming blue on exposure.	Black or red coating of copper.	White.	Solution of suboxide of copper in am. becomes blue on exposure.
Greenish white, sol. in am.	Ditto.	White, sol. in ex.	Organic bodies impede pptn. by potash; characteristic tests, am., blowpipe and metallic iron, which ppts. copper.
0	Brown, of reduced gold.	Yellowish green.	Protochloride of tin gives purple ppt.; protosulph. iron and sulphurous acid give metallic gold.
Slow discoloration.	Black powder. (iridium.)	Discoloration.	

Behaviour of Salts of THIRD CLASS

	Potash.	Carbonate of Potash.	Bicarbonate of Potash.
Lead, oxide of.	White, sol. in ex.	White, insol. in ex., sol. in potash.	White, insol. in ex., sol. in potash.
Mercury, suboxide of.	Black, insol. in ex.	Yellow, becomes black on boiling.	White, blackens on boiling.
Mercury, protoxide of.	Yellow, insol. in ex., white with mur. am.	Reddish brown ; white with mur. am.	Brown red.
Molybdous oxide.	Brown black, insol. in ex.	Brown black, slightly sol. in ex.	Brown black, slightly sol. in ex.
Molybdic oxide.	Bulky brown black, insol. in ex.	Brown, slightly sol. in ex.	Brown, sol. in ex.
Osmic oxide.	Black on boiling or on standing.	Black on standing.	Black on boiling.
Palladious oxide.	Brownish yellow, sol. in ex.	Brownish, sol. in ex., reprecipitated on boiling.	Brownish, sol. in ex.

OF OXIDES *with Reagents.* B. No. 1.

Ammonia.	Carbonate of Ammonia.	Yellow Pruss. of Potash.	Red Prussiate of Potash.
White, insol. in ex. (slowly formed in acetate).	White, insol. in ex.; sol. in potash.	White.	0
Black, insol. in ex.	Grey or black.	Gelat. white.	Reddish brown.
White, insol. in ex.	White.	White.	Yellow in nitrate or sulphate; 0 in chloride.
Brown black, insol. in ex.	Brown black, sol. in ex.	Reddish brown.	Reddish brown.
Bulky brown black, insol. in ex.	Brown, sol. in ex.	Brown.	Brown.
Brown on standing.	Brown on standing.	0	0
Yellowish brown, sol. in ex.	Decolorises the solution.	Gelat. green, formed slowly.	Gelat. on standing.

Behaviour of Salts of THIRD CLASS

	Hydrosulphate of Ammonia.	Sulphuretted Hydrogen.	Oxalic Acid.
Lead, oxide of.	Black, insol. in ex.	Black.	White.
Mercury, suboxide of.	Black, insol. in ex.	Black, insol. in NO_5 .	White.
Mercury, protoxide of.	Black; insol. in ex.; sol. in pot.	Black, insol. in NO_5 .	White, in nitrate or sulphate. 0 in chloride.
Molybdous oxide.	Brown yellow, sol. in ex.	Brown black, formed slowly.	0
Molybdic oxide.	Brownish yellow, sol. in ex.	Brown, formed slowly.	0
Osmic oxide.	Brownish yellow, insol. in ex.	Brownish yellow, formed slowly.	0
Palladious oxide.	Black, insol. in ex.	Black.	0

OF OXIDES *with Reagents.* B. No. 2.

Phosphate of Soda.	Bar of Metallic Zinc.	Iodide of Potassium.	Remarks.
White, sol. in potash.	Reduced lead.	Yellow, sol. in ex.	SO ₃ , a white ppt. HCl. in a strong sol. a white ppt. Chromate of potash, a yellow ppt., sol. in pot.
White.	Mercury reduced.	Greenish yellow, becoming black, sol. in ex.	HCl. a white ppt. blackened by am. Chromate of pot. a red ppt.
White, in nitrate or sulphate. A faint troubling in chloride.	Mercury reduced.	First yellow, then scarlet, sol. in ex., sol. in HCl.	Protochloride of tin in excess gives metallic mercury. In a galvanic circle, formed by a drop of sol. of mercury on gold, touched with a piece of iron, mercury is deposited on the gold.
Brown black.			
Brownish white.			
Black, formed slowly.	Black powder of reduced osmium.	Black after standing.	Free chloride of osmium is decomposed by water into osmic acid and osmium, which is pptd.
Brown.	Black powder of reduced palladium.	Black.	Cyanide of mercury gives a yellowish white ppt. of cyanide of palladium, sol. in HCl.

Behaviour of Salts of THIRD CLASS

	Potash.	Carbonate of Potash.	Bicarbonate of Potash.
Platinous oxide.	0	Brown, slowly formed.	0
Platinic oxide.	Yellow, sol. in ex. if hot ; insol. in HCl.	Yellow, insol. in ex.	Yellow, insol. in ex.
Rhodic oxide.	On ebullition, gelat. brownish yellow.	Yellowish, formed slowly.	
Silver, oxide of.	Brown, insol. in ex., sol. in am.	White, sol. in am.	White, sol. in am.
Telluric oxide.	White, sol. in ex.	White, sol. in ex.	White, sol. in ex.
Tin, protoxide of.	White, sol. in ex., soln. gives metallic tin on heating.	White, insol. in ex.	White, insol. in ex.
Tin, peroxide of.	White, sol. in ex.	White, effervescence ; sol. in ex.	White, effervescence ; insol. in ex.

OF OXIDES *with Reagents.* C. No. 1.

Ammonia.	Carbonate of Ammonia.	Yellow Prussiate of Potash.	Red Prussiate of Potash.
Green, cryst.	0	0	0
Yellow, sol. in ex. if hot ; insol. in alkalies.	Yellow, insol. in ex.	Yellow, insol. in acids, sol. in hot alkalies.	Yellow, insol. in acids, sol. in hot alkalies.
Yellowish, sol. in HCl.	Yellowish, formed slowly.	0	0
Brown, very sol. in ex.	White, sol. in ex.	White.	Reddish brown.
White, sol. in ex.	White, sol. in ex.	0	0
White, insol. in ex.	White, insol. in ex.	White, gelat.	White.
White, sol. in ex.	White, effervescence ; insol. in ex.	Yellowish, gelat. on standing.	0

Behaviour of Salts of THIRD CLASS

	Hydrosulphate of Ammonia.	Sulphuretted Hydrogen.	Oxalic Acid.
Platinous oxide.	Dark brown, sol. in large ex.	Black, formed slowly, slight- ly sol. in ex.	0
Platinic oxide.	Brownish black, sol. in ex.	Brown, formed slowly.	0
Rhodic oxide.	Brown, insol. in ex.	Brown, formed slowly.	0
Silver, oxide of.	Black, insol. in ex.	Black.	White, sol. in am.
Telluric oxide.	Brown, sol. in ex.	Brown.	0
Tin, prot- oxide of.	Brown, sol. in ex.	Brown.	White.
Tin, per- oxide of.	Yellow, sol. in ex.	Yellow, in acid sol.	0

OF OXIDES *with Reagents.* C. No. 2.

Phosphate of Soda.	Bar of Metallic Zinc.	Iodide of Potassium.	Remarks.
0	Black powder of reduced platinum.	Black, metallic appearance, formed slowly.	Behaviour with am. is characteristic.
0	Ditto.	Brown.	Behaviour with potash and am. is characteristic. Chloride of potassium and mur. am. give yellow ppts.
0	Metallic rhodium.	Dark ppt. formed slowly.	Salts have mostly a red colour.
Yellow, sol. in am.	Metallic silver.	Yellowish white, sol. in ex., insol. in am., insol. in NO_5 .	HCl. gives a white curdy ppt. of chloride of silver, insol. in acids, very sol. in am. (characteristic.)
White.	Tellurium, bulky black.		(See Blowpipe Tables.)
White.	Metallic tin, grey.	Yellowish white, sol. in ex., sol. in HCl.	Chloride of gold gives a blue ppt. (purple of Cassius.)
White.	White, gelat., perox. of tin, hydrogen evolved.	0	Behaviour with sulphuretted hydrogen and hydrosulph. am. is characteristic.

Behaviour of THIRD CLASS OF OXIDES

Acids (in combination).	Nitrate of Silver.	Chloride of Barium.
Antimonic.	White.	White.
Antimonious.	White.	White, slightly sol. in water.
Arsenic.	Chocolate red, sol. in nitric acid, sol. in am.	White, sol. in acids, sol. in mur. am.
Arsenious.	Pale yellow, sol. in acetic acid, sol. in am. ; the ammonio-nitrate of silver gives the same ppt. in free acid.	0
Chromic.	Reddish brown.	Canary yellow, sol. in NO_5 .
Molybdic.	White, sol. in water, nitric acid, and am.	White, sol. in water ; sol. in NO_5 .
Hyper-Manganic.	A yellowish brown precipitate.	0
Manganic.	Black, of perox. of manganese.	0
Osmic.	Brown.	Brown.
Selenious.	White, slightly sol. in NO_5 .	White, sol. in HCl .
Tungstic.	White.	White.
Vanadic.	Bulky yellow, sol. in NO_5 , and in am. Bleaches on exposure.	Bulky orange yellow. Bleaches on exposure.

with Reagents. Acids. D. No. 1.

Acetate of Lead.	Protonitrate of Mercury.	Nitrate of Lime.
White.	White.	White.
White.	White.	White, slightly sol. in water.
White, sol. in NO_5 , insol. in acetic acid.	Yellowish white, sol. in NO_5 .	White, sol. in HCl , in mur. am. and in acetic acid.
White, sol. in NO_5 and in acetic acid.	Yellowish white, sol. in NO_5 .	0
Yellow, sol. in alkalis.	Yellowish red.	0
White, sol. in NO_5 .	Yellowish, sol. in NO_5 .	White, sol. in water, in HCl . and in NO_5 .
A ppt. of ox. of lead (carbonate?), and of perox. manganese.	Ppt. of perox. manganese.	0
Ditto.	Ditto.	Perox. manganese.
Brown, with basic acetate of lead.	Brown, sol. in NO_5 .	Brown.
White, slightly sol. in NO_5 .	White, sol. in NO_5 .	White, sol. in NO_5 .
White.	White.	White.
Yellow, sol. in water.	Yellow.	0

Behaviour of THIRD CLASS OF OXIDES

Acids (in combination).	Sulphuretted Hydrogen (in acid soln).	Hydrosulphate of Ammonia.
Antimonic.	Orange yellow, sol. in alkalis.	Orange yellow, sol. in ex.
Antimonious.	Ditto.	Ditto.
Arsenic.	Yellow, sol. in alkalis.	Yellow, on adding an acid.
Arsenious.	Ditto; slightly sol. in pure water.	Ditto.
Chromic.	Chromic acid reduced to oxide of chromium.	Greenish (oxide of chromium).
Molybdic.	Brown.	Brown on adding an acid.
Hyper-Manganic.	In neutral soln. flesh colour.	Flesh colour, insol. in ex.
Manganic.	Ditto.	Ditto.
Osmic.	Brown black.	Black, insol. in ex.
Selenious.	Citron yellow, sol. in hydrosulph. am.	Yellow, sol. in ex.
Tungstic.	Slight turbidity.	Brown with very dilute HCl.
Vanadic.	Grey.	Sol. becomes brown.

with Reagents. Acids. D. No. 2.

Rod of Metallic Zinc.	Hydrochloric Acid.	Remarks.
With HCl. a black powder (antimony).	White (antimonious acid).	
Ditto.	White (antimonious acid).	
With HCl. gives metallic arsenic.	0	(See Section on Detection of Poisons.)
Ditto.	0	Sulphate of copper gives a green precipitate. Ammonio-sulphate of copper gives a green ppt. in free acid.
0 With sulphuric acid, chromic acid becomes oxide.	Chlorine evolved.	Organic bodies reduce chromic acid to oxide of chromium.
Dark brown with HCl.	0 (or sol. in ex.)	
Perox. Manganese.	Chlorine evolved.	Sol. decolorized by sulphurous acid.
Ditto.	Ditto.	Ditto.
With HCl., reduced osmium.	Free osmic acid, known by its odour.	Sulphurous acid and organic bodies slowly reduce osmic acid.
With HCl., reduced selenium.	0	Sulphurous acid gives a floccy red ppt. of reduced selenium.
With HCl. or SO_3 a fine blue colour.	White, insol. in ex.	
0	Chlorine evolved.	Sulphurous acid and organic bodies reduce vanadic acid to oxide of vanadium.

Behaviour of ACIDS with Reagents

Acids (neutralized).	Chloride of Barium.	Nitrate of Lime.	Acetate of Lead.	Nitrate of Silver.
Acetic.	0	0	0	0 A cryst. ppt. if solns. be conctd.
Boracic.	White, sol. in ex., in acetic acid, and in mur. am.	White, sol. in ex., sol. in mur. am.	White, sol. in acetic acid.	White, sol. in acetic acid.
Bromic.	White, sol. in water.	0	White, sol. water.	White, sol. in NO ₃ .
Carbonic.	White.	White.	White.	Brownish white, sol. in am.
Chloric.	0	0	0	0
Citric.	White in in strong solns.	White, slightly sol. in water.	White.	White.
Formic.	0	0	0 Except in very strong so- lutions.	Heated slowly, a black pow- der of me- tallic silver.

(*Excess neutralized by an Alkali*). A.

Protonitrate of Mercury.	Sulphuric Acid (concentrated).	Remarks.
0 A cryst. ppt. if solns. be conctd.	Acetates moistened with SO_3 evolve acetic acid, known by its pungent odour.	
Yellowish white, sol. in ex., sol. in NO_5 .	Borates moistened with SO_3 and then with alcohol, cause the latter to burn with a green flame.	
Yellow, sol. in NO_5 .	Bromine is evolved, sol. becoming yellow.	Bromates mixed with combustibles detonate when heated, or when moistened with oil of vitriol.
Light brown or black.	Effervescence of carb. acid, in-odorous.	Carbonates effervesce with acids.
0	Peroxide of chlorine evolved ; liquid bleaches vegetable colours.	
White.		The ppt. by a citrate in a salt of lead, when washed, dissolves in ammonia.
0 When heated, mercury is reduced to the metallic state.	Evolves formic acid, known by its pungent odour.	Characteristic test is heating with nitrate of silver.

Behaviour of ACIDS with Reagents

Acids (neutralized).	Chloride of Barium.	Nitrate of Lime.	Acetate of Lead.
Hydrochloric.	0	0	White, slightly sol. in water.
Hydrocyanic.	0 If moderately di- lute.	0	White.
Hydrobromic.	0	0	White, sol. in NO ₅ .
Hydrofluoric.	White, sol. in HCl.	Bulky white.	White, sol. in HCl.
Hydriodic.	0	0	Yellow, sol. in NO ₅ .
Hydroselenic.	0	0	Black.
Hydrosulpho- cyanic.	0	0	0 if moderately di- lute.
Hydrosul- phuric.	0	0	Black.

(*Excess neutralized by an Alkali*). B.

Nitrate of Silver.	Protonitrate of Mercury.	Sulphuric Acid (concentrated).	Remarks.
Curdy white, insol. in NO_5 , sol. in am.	White, insol. in cold NO_5 blackened by am.	Evolves HCl.	Nitrate of silver with nitric acid is a characteristic test.
White, insol. in dilute NO_5 ; sol. in am. and in cyanide of potm.	Grey, reduced mercury.	Evolves hydrocyanic acid.	Pppte a mixture of proto and per-salt of iron by potash, add a sol. cyanide, and treat with HCl. Pruss. blue remains. (Scheele's test.)
Yellow, insol. in NO_5 ; sol. in am.	Yellow, insol. in cold NO_5 .	Bromine is evolved.	Chlorine water sets bromine free, soln. becoming brown; colour removed by agitation with ether.
0	0	Hydrofluoric acid evolved, ascertained by its corroding glass.	
Yellow, insol. in NO_5 , nearly insol. in am.	Yellowish green, insol. in NO_5 .	Iodine evolved.	Chlorine water added gradually renders iodine free, ascertained by starch, which gives a blue colour.
Black.	Black.	Seleniatted hydrogen evolved; odour of decayed cabbage.	Odour produced before the blowpipe is the most characteristic test.
Curdy white, sol. in strong but not in dilute am.	Grey, reduced mercury.	Free hydrosulphocyanic acid; deposits a yellow powder.	Gives a red colour with persalts of iron, destroyed by chloride of mercury.
Black, insol. in am.	Black, insol. in NO_5 .	Sulphuretted hydrogen disengaged.	Fœtid odour of free gas destroyed by Cl. with deposition of sulphur.

Behaviour of ACIDS with Reagents

Acids (neutralized).	Chloride of Barium.	Nitrate of Lime.	Acetate of Lead.	Nitrate of silver.
Hyposulphuric.	0	0	0	0
Hyposulphurous.	0 unless concentrated.	0	White, slightly sol. in NO_5 .	White, becomes black.
Iodic.	White, sol. in NO_5 .	White, sol. in NO_5 .	White, sol. in NO_5 .	White, sol. in NO_5 and in am.
Nitric.	0	0	0	0
Nitrous.	0	0	0	White, slightly sol. in water.
Oxalic.	White, nearly insol. in water, sol. in acids.	White, insol. in water and dilute acids.	White.	White.
Perchloric.	0	0	0	0

(Excess neutralized by an Alkali). C.

Protonitrate of Mercury.	Sulphuric Acid (concentrated).	Remarks.
0	Sulphurous acid evolved; no ppt. of sulphur.	HCl. evolves sulphurous acid, and sulphuric acid remains, ascertained by a salt of barytes.
Black, insol. in NO_5 .	Sulphurous acid disengaged, and free sulphur deposited.	Soluble hyposulphites dissolve chloride of silver, forming a sweet solution.
White, sol. in NO_5 .		Sulphurous acid develops free iodine, indicated by starch, which gives a blue colour.
0	With copper filings nitric oxide is evolved.	Free nitric acid with HCl. dissolves gold. Bleaches indigo, if hot. A nitrate, heated with protosulph. iron and oil of vitriol, gives a dark brown colour.
0	Nitric oxide and nitric acid formed.	With HCl. does not dissolve gold.
White.	Heated with sulphuric acid, a dry oxalate gives carbonic oxide and carbonic acid gases, the first is inflammable, the last is detected by causing lime-water to become milky. Sol. of oxalic acid ppts. a soln. of sulphate of lime.	
0	The free acid pptes. salts of potash in strong solutions. When heated to redness, perchlorates evolve oxygen gas, and leave chlorides.	

Behaviour of ACIDS with Reagents

Acids (neutralized).	Chloride of Barium.	Nitrate of Lime.	Acetate of Lead.
Phosphoric (tribasic).	White, sol. in HCl. or mur. am. in ex.	White, sol. in NO ₅ .	White, sol. in NO ₅ , insol. in acetic acid.
Phosphorous.	White, sol. in HCl. sol. in mur. am.	White, sol. in NO ₅ .	White, sol. in NO ₅ , insol. in acetic acid.
Selenic.	White, insol. in NO ₅ .	White, sol. in NO ₅ .	White, insol. in NO ₅ .
Silicic.	White, sol. in dilute HCl.	White, sol. in dilute NO ₅ .	White, sol. in NO ₅ , sol. in potash.
Sulphuric.	White, insol. in acids.	White, sol. in water.	White, slightly sol. in NO ₅ .
Sulphurous.	White, sol. in HCl.	White, sol. in water.	White, sol. in dilute NO ₅ .
Tartaric.	White, slightly sol. in ex.; more sol. in dilute acids.	White, almost insol. in water.	White.

(*Excess neutralized by an Alkali*). D.

Nitrate of Silver.	Protornitrate of Mercury.	Sulphuric Acid.	Remarks.
Yellow, sol. in NO_5 , sol. in am.	White, sol. in NO_5 .		Sol. of magnesia with am. gives a white cryst. ppt.*
Brown, reduced silver.	Grey, reduced mercury.		Characteristic property is power of reducing salts of silver and mercury.
White, sol. in NO_5 .	0	Seleniates mixed in the solid state with mur. am. and heated, give selenium. Seleniates boiled with HCl. evolve Cl., and sulphuretted hydrogen then causes a ppt.	
White, sol. in dilute NO_5 .	Yellowish white, sol. in dilute NO_5 .	Produces a jelly with hydrochloric or sulphuric acid, or leaves, on evaporating the mixture to dryness, a residue insol. in water.	
0 White, in a very con- ctd. soln.	White, sol. in NO_5 .		A salt of barytes is a characteristic test.
White, sol. in NO_5 , sol. in am.	Grey, reduced mercury.	Sulphurous acid is evolved.	Decomposes iodic acid, giving free iodine, ascertained by starch.
White, sol. in am.	White, sol. in NO_5 .		The sol. tartrates mixed with bisulphate of pot. cause a ppt. of bitartrate of pot., sol. in HCl. and alkalies.

* In a salt soluble in water, phosphoric acid is easily recognized by the yellow precipitate by nitrate of silver in neutral solutions; if the substance is insoluble in water, it is better to fuse it with an alkaline carbonate, when a phosphate of the alkali is formed, which together with the excess of carbonate, dissolves when the mass is treated with water. When made neutral by an acid, this solution may be tested with nitrate of silver. (See also Schulze's method of estimating phosphoric acid in quantitative analysis.)

CHAPTER III.

QUALITATIVE ANALYSIS.

THE performance of a satisfactory qualitative analysis of a complex substance by the aid of the preceding tables, would be a matter of some difficulty in their present arrangement. Instead of applying each reagent successively, the method adopted in examining the composition of single or mixed bodies, consists in determining the presence or absence of certain *classes* of substances by the application of a single test for each class ; these classes can be divided and subdivided by other tests, and the subdivisions examined for their individual members. A particular class of metals is, for instance, precipitated by sulphuretted hydrogen ; by the behaviour of this class with hydrosulphate of ammonia, it admits of a farther division, some of the metals precipitated by sulphuretted hydrogen being soluble, the remainder insoluble, in that liquid ; thus affording two divisions, each of which, if both are found to exist, is then examined for its individual members.

The advantage of this method of procedure arises from its being in general easier to discover classes than particular bodies ; and the absence of a class being demonstrated, it is of course unnecessary to examine by other tests for any particular member. The principal feature in this system is, therefore, that of exclusion ; on the application of a test, hydrosulphate of ammonia, for example, when we perceive none of the appearances which that test should produce with certain metals, we reject these as absent from the combination. We thus have a negative result ; but to prove the absence of particular substances is in itself just as important in qualitative analysis as to demonstrate their presence.

A substance may occasionally be presented for examina-

tion, whose composition can be very well ascertained by the application of two or three reagents; but it is advisable, and will mostly, in the end, be found more satisfactory, especially when we are not assured of the purity of the substance under examination, to follow some general rule, or particular system, rather than to try the effect of a reagent at random, from some vague idea we had before entertained of the composition of the substance. Such systems are developed in the following tables, where, it will be seen, classes are formed, divided, subdivided, and, if necessary, divided further, characters being described by which the particular members of each subdivision are distinguished from one another.

An examination of the physical characters of a substance must always precede its analysis. It is of importance to know its density, volatility, combustibility, and solubility. Before proceeding to determine the inorganic constituents of a substance, it is also necessary to ascertain whether or not any organic matter is present, this materially affecting many metals in their behaviour with reagents, as may be seen by the preceding tables. Almost all fixed organic bodies possess the property of becoming blackened or carbonized by heating out of contact with the air. To discover organic matter, therefore, a small portion of the substance should be heated in a glass tube, free from lead, when, in general, blackening will occur, and an empyreumatic odour be developed, if organic matter is present. The tube should be held slightly inclined, and be heated by a spirit lamp.

There are, however, a few inorganic bodies which become black on heating; and, on the contrary, some organic bodies which do not blacken. But there is another character of organic bodies, which, taken with the blackening by heat, may be considered decisive. This is the property they all possess of deflagrating when thrown into fused nitre; a property possessed, too, by a few inorganic bodies, but by none which also blacken on heating. The two tests conjoined are therefore conclusive.

By the same operation of heating in a tube to discover organic matter, we ascertain (when heated gradually) whether the substance contains *water*. If so, this condenses on the sides of the glass tube before the heat is sufficient to carbonize the substance. If the condensed water is alkaline, this shows the presence of *ammonia*.

If organic matter is present, and the object of the analysis is merely to discover the fixed bases, and not the acids of the substance, it should be destroyed by combustion in the open air, either in a platinum or a porcelain * crucible, uncovered, heated very strongly by a spirit lamp with circular wick, or by the inflammable mixture of gas and air described page 10.

The proper solvent of the substance must next be sought. Pure water is first to be tried; if neither wholly nor partially soluble in this, hot hydrochloric or nitric acid or aqua regia must be tried successively, until it dissolves entirely, or leaving only a slight residue, which must be collected and examined by other means. Acids frequently leave insoluble residues, which have been formed by their action on the substance to be dissolved, and did not therefore pre-exist in the substance under examination: these may be easily distinguished in appearance from the original substance; thus silica remains as a jelly when some silicates are treated with acids; sulphur when sulphurets, and selenium when seleniurets are acted on by nitric acid. In the last two cases, sulphur and selenium are slowly dissolved by the further action of the acid, sulphuric and selenic acids being formed.

The course of qualitative analysis to be followed differs somewhat for substances soluble and insoluble in water; but the following tables have been so arranged as to include the necessary modifications for both. When insoluble, both in water and acids (as is the case with most silicates), the substance must be rendered soluble in acids by fusion with an

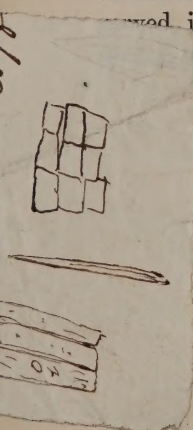
* When the substance can contain metals reducible by charcoal, and which would alloy with platinum at a red heat, a porcelain crucible must be used; in other cases platinum is preferable.

alkaline carbonate. This operation consists, 1. in reducing the substance to a state of extremely minute division in an agate mortar; 2. mixing with twice or thrice its weight of pure carbonate of soda or potash; and 3. fusing in a covered platinum crucible. The resulting mass is then treated with pure hydrochloric or nitric acid, and is in almost every case dissolved, leaving, in the case of silicates, a gelatinous residue of silicic acid. (See the Analysis of Silicates for details of this operation.) When fusion with carbonate of soda is inadequate, caustic potash (*potassa fusa*) is to be substituted, and the fusion performed with care in a silver instead of a platinum crucible.

The first section of this chapter contains directions for the qualitative analysis of a salt consisting only of a single acid and a single base, comprising all those which are of ordinary occurrence: with such, rather than with complex substances, the beginner will do well to exercise himself.

Respecting the quantity of matter to be operated on, it will be found more convenient to work with small than with large amounts; from ten to twenty grains will be sufficient in almost every case. A small quantity must always be reserved in case of accidents, or to examine the action of any
est.

LIST OF BASES TO BE SOUGHT FOR.

- 
- Class 1. {
- Antimony, oxide of
 - Bismuth, oxide of
 - Cadmium, oxide of
 - Copper, protoxide of
 - Gold, oxide of
 - Iron, peroxide of
 - Lead, oxide of
 - Mercury, protoxide of
 - Mercury, suboxide of
 - Silver, oxide of
 - Tin, peroxide of
 - Tin, protoxide of.

Class 2.	{	Alumina
		Chromium, oxide of
		Cobalt, oxide of
		Iron, protoxide of
		Manganese, protoxide of
		Nickel, oxide of
Class 3.	{	Zinc, oxide of.
		Barytes
		Lime
		Magnesia
Class 4.	{	Strontian.
		Ammonia
		Lithia
		Potash
		Soda.

LIST OF ACIDS TO BE SOUGHT.

Arsenic	Hydrosulphuric
Boracic	Iodic
Carbonic	Nitric
Chloric	Phosphoric
Hydrochloric	Sulphuric
Hydrofluoric	Sulphurous.
Hydriodic	

SECTION I.

QUALITATIVE ANALYSIS OF A SALT CONSISTING OF A SINGLE ACID AND A SINGLE BASE, CONTAINED IN THE PRECEDING LIST.

Solution made in water, hydrochloric or nitric acid, aqua regia, or by fusion with carbonate of soda and solution in hydrochloric acid.

I. EXAMINATION FOR BASE.

According to the following table, in an examination for the base of a single salt, the first operations have for their object the assignment of the base to one of three classes, named A, B, and C; A being class 1., B class 2., and C classes 3. and 4., of the preceding list. In the table, it is directed

that (1.) sulphuretted hydrogen gas be passed through the solution of the substance made slightly acid. If a precipitate appears, the base is then among the list forming class 1., and may be determined by tests given under A page 61. If the base is not precipitated by sulphuretted hydrogen (2.), hydrosulphate of ammonia is applied to the solution previously made alkaline. The production of a precipitate by this reagent shows the base to be among the list forming class 2., and may be determined by tests described under B page 61. If neither sulphuretted hydrogen nor hydrosulphate of ammonia produces a precipitate (3.), the base is one contained in classes 3. and 4., and may be determined by operations described under C page 62.

Frequent advantage will be taken of a tabular method of exhibiting at one view the entire course of an analysis by means of brackets, under the opposite arms of which will be mentioned the two results which can be afforded by the application of a reagent, namely, the precipitate and the remaining solution. The precipitate is mentioned at the left hand, and the remaining solution at the right hand arm of the bracket. In the analysis of mixed compounds the precipitate and the solution are generally separated by filtration, and each examined apart by other tests, the results which can be produced by these being, as before, mentioned under the opposite arms of brackets. This plan may be readily conceived by reference to the table, page 66.

I. EXAMINATION FOR BASE.

If not already acid, a portion of the solution must be made slightly so, by hydrochloric, unless this causes a white precipitate of chloride of silver, subchloride of mercury, or chloride of lead, when very dilute nitric acid must be used.

Transmit sulphuretted hydrogen gas through the solution.

Precipitate can contain the sulphuret of one of the following metals :

Cadmium
Lead
Bismuth
Copper
Silver

together with sulphur, caused by a persalt of iron, and sulphuret of arsenic from arsenious or arsenic acid.

(A.)

If the base remain in solution, it may be either oxide of chromium, nickel, cobalt, zinc, protoxide of iron, protox. of manganese, alumina, magnesia, lime, strontian, barytes, ammonia, soda, potash, or lithia. Render the solution alkaline by ammonia, and then add a slight excess of hydrosulphate of ammonia.

Precipitate may contain sulphuret of manganese, zinc, cobalt, nickel, or iron ; alumina or ox. of chromium ; and if the substance under examination was insoluble in water, it may contain magnesia, lime, strontian, or barytes, united with either phosphoric, arsenic, or boracic acid, or their metallic bases with fluorine.

(B.)

If hydrosulphate of ammonia produces no precipitate, the solution may contain —

Magnesia
Strontian
Lime
Barytes
Lithia
Soda
Potash
Ammonia.

(C.)

(A.) *If a precipitate be formed by sulphuretted hydrogen.*

(Class 1. page 57.)

If the colour of the precipitated sulphuret be dark brown or black.		<i>Tests to be applied to the original solution :</i>	
		1. Lead.	{ A solution of a sulphate gives a white precipitate of sulphate of lead.
		2. Bismuth.	{ The saturated solution of the substance becomes milky on dilution with water, if the base be bismuth.
		3. Copper.	{ Ammonia gives an intense blue colour.
		4. Silver.	{ Hydrochloric acid, a white precipitate ; add ammonia in excess to the precipitate — if
		5. Subox. of mercury.	{ chloride of <i>silver</i> , it is dissolved ; if subchloride of <i>mercury</i> , it becomes black.
		6. Protox. of mercury.	{ Caustic potash, a yellow precipitate of oxide. Iodide of potassium gives a yellow precipitate, becoming scarlet.
		7. Gold.	{ Protochloride of tin, a purple precipitate.
If orange red.		8. Protox. of tin.	{ Chloride of gold, a purple precipitate.
		9. Oxide of antimony.	{ Filter and dry the precipitated sulphuret, and dissolve it in a little hydrochloric acid ; evaporate off most of the excess of acid, and pour the remaining solution into water : if antimony, a white precipitate of oxichloride is formed.
If yellow.	{	10. Cadmium.	{ If the sulphuret be insoluble in hydrosulphate of ammonia.
		11. Perox. of tin. (Arsenic.)	{ If the sulphuret be soluble in hydrosulphate of ammonia. If <i>arsenic</i> , the precipitated sulphuret is volatile at a high temperature ; dried, mixed with black flux, and heated to redness in a glass tube, it gives a sublimate of metallic arsenic. If not volatile, and no sublimate of metallic arsenic be produced, the base is perox. of tin.

Note. — A milk-white precipitate of sulphur may be produced from the decomposition of sulphuretted hydrogen by a salt of the peroxide of iron.

(B.) *If no precipitate be produced by sulphuretted hydrogen, but one by hydrosulph. of Ammonia.* (Class 2. page 58.)

If black.	{	1. Nickel.	{ Carbonate of soda gives, with the original solution, an apple-green precipitate.
		2. Cobalt.	{ Carbonate of soda, a dull red precipitate.
		3. Protox. of iron.	{ Carbonate of soda, a green precipitate, becoming red on exposure to the air.

Note. — Protox. of iron might have existed originally as peroxide, being reduced by sulphuretted hydrogen. To discover this, apply the tests of red and yellow prussiates of potash to the original solution.

If *dull green*.—4. Chromium. Confirm by colour of bead produced with microcosmic salt before the blowpipe. (See Blowpipe Tables.)

If *flesh colour*.—5. Manganese. Confirm by colour of bead produced with microcosmic salt before the blowpipe; exterior and interior flames.

If *white*. {

6. Alumina.	{	Ammonia gives, with the original solution, a white precipitate, insoluble in excess, but soluble in caustic potash.
7. Oxide of zinc.		Ammonia gives, with the original solution, a white precipitate, soluble in excess.

If the white precipitate be neither alumina nor oxide of zinc, and the substance under examination be insoluble in water, it may have for base lime, magnesia, strontian, or barytes, united to certain acids mentioned in page 60. If so, the solution of the substance in an acid is treated in the manner described below for the detection of these same earths, when *no* precipitate is caused by hydrosulphate of ammonia.

(C.) If neither sulphuretted hydrogen nor hydrosulphate of ammonia produce a precipitate. (Classes 3. and 4. page 58.)

The solution may contain either lime, magnesia, strontian, barytes, lithia, soda, potash, or ammonia.

To a portion of the solution add carbonate of soda.

Class 3. is precipitated.	Class 4. is not pre-
To the concentrated original solution add sulphuric acid.	cipitated.

Barytes, lime, or strontian may be precipitated.

Add solution of sulphate of lime to the undiluted solution of the substance.

If no precipitate, the base is *magnesia*. Confirm by applying the test of phosphate of soda with ammonia.

Barytes (sulphate) is precipitated.

If no immediate precipitate be produced by sulphate of lime, the solution may contain lime or strontian.

To the original solution somewhat diluted, add a few drops of a solution of a sulphate: *Strontian* is precipitated, *Lime* is not.

If carbonate of soda produces no precipitate, the base is among Class 4. p. 58.

Potash.	{ A solution of tartaric acid in alcohol gives a white crystalline precipitate of bitartrate of potash, with the concentrated original solution.
Ammonia.	{ Boiled with caustic potash, the odour of ammonia is evolved, and a rod, moistened with hydrochloric acid, held over the vessel, produces white fumes.
Lithia.	{ Phosphate of soda, to which ammonia has been added, gives, on agitation and standing, a white precipitate. Salts of lithia communicate a crimson colour to the flame of the blowpipe.
Soda.	{ If none of these characters is observed, and the salt tinges the blowpipe flame yellow, the base is soda.

Note. — If fusion with an alkaline carbonate be necessary to effect the solution of the substance, when alkalies are sought, carbonate of barytes or of lime should be substituted for carbonate of soda. The barytes or lime may be removed from the solution of the ignited mass in an acid, by carbonate of ammonia as an insoluble carbonate; the filtered solution is then evaporated, the residue calcined to expel ammoniacal salts, and dissolved in water. The solution thus obtained is tested for potash by tartaric acid or chloride of platinum, or for lithia or soda by the blowpipe.

2. EXAMINATION FOR ACID.

If the substance has acid properties, neutralize with an alkali; if alkaline, neutralize with nitric acid.

To the concentrated solution of the salt in water, if soluble, or to the dry salt, if insoluble, add dilute sulphuric acid.

If sulphuric acid produces neither effervescence nor free iodine, add solution of nitrate of barytes to the neutral solution of the salt.

If *effervescence* occurs, the acid may be either *carbonic*, *sulphuric*, or *hydrosulphuric*. If iodine be set free (ascertained by starch), the acid is *hydriodic*. (A.)

Not precipitated : —
hydrochloric, *hydriodic*, *nitric*, *chloric*.
(C.)

Precipitated : — *sulphuric*, *phosphoric*, *arsenic*, *boracic*, *hydrofluoric*, *iodic*. (B.)

(A.)

If effervescence on adding dilute sulphuric acid.

Carbonic.	{ The gas is inodorous, and when passed through lime-water, causes a white precipitate, which dissolves when an excess of the gas is passed.
Sulphurous.	{ Evolved gas has the pungent smell of burning sulphur.
Hydrosulphuric.	{ The gas has a fœtid odour, and the solution of the salt produces a black precipitate in salts of lead.

To detect hydriodic acid (iodides), mix the solution of the salt with a little solution of starch, and allow chlorine gas to fall on the surface of the liquid: the blue iodide of starch is formed.

(B.)

Acids precipitated by nitrate of barytes, when no effervescence occurs with sulphuric acid.

If the precip. by nitrate of barytes be soluble in hydrochloric acid.	Sulphuric.	{ The precipitate is insoluble in hydrochloric acid.
	Hydrofluoric.	{ The powdered salt, gently heated with oil of vitriol in a platinum crucible, evolves a vapour which corrodes glass.
	Arsenic (arsenious).	{ Sulphuretted hydrogen passed through the solution of the substance, slightly acidified by hydrochloric acid, gives a yellow precipitate of sulphuret of arsenic. This dried, mixed with black flux, and heated in a glass tube, gives a sublimate of metallic arsenic.
	Phosphoric.	{ Nitrate of silver gives, with the neutral solution of the salt, a yellow precipitate of phosphate of silver, soluble in nitric acid.
	Boracic.	{ The dry salt, moistened with oil of vitriol, and then alcohol added, the latter burns with a green flame.
	Iodic.	{ Sulphurous acid developes iodine in the original solution: ascertain this by starch.

(C.)

If dilute sulphuric acid produces no effervescence, and nitrate of barytes no precipitate, the acid may be hydrochloric, hydriodic, nitric, or chloric.

Add nitrate of silver to the solution.

If no precipitate be produced, the acid is either *nitric* or *chloric*.

To detect nitric, add the dry salt to a mixture of protosulph. of iron and oil of vitriol; immediately, or on heating, a brown colour is developed, owing to nitric oxide.

To detect chloric, add oil of vitriol to the dry salt: peroxide of chlorine is evolved, and the liquid bleaches vegetable colours.

If a yellow precipitate, *hydriodic* (iodide of silver), insoluble in ammonia.

If a white curdy precipitate, *hydrochloric* (chloride of silver), soluble in ammonia.

Note.— If the substance under examination be a metallic *sulphuret*, when dissolved by means of nitric acid or aqua regia, the sulphur becomes converted into *sulphuric acid*.

SECTION II.

QUALITATIVE ANALYSIS OF A COMPOUND WHICH CAN CONTAIN ALL THE BASES AND ACIDS IN THE LIST, PAGES 57 AND 58.

Solution made in water, hydrochloric or nitric acid, or by fusion with carbonate of soda, and subsequent solution in hydrochloric acid.

With mixed substances, the examination is to be conducted as if the compound really contained all the ingredients of which it might possibly be composed; and when a reagent does not produce a precipitate, the solution is then to be treated in the same manner as if a precipitate had been formed and separated from the liquid by filtration. It is of the highest importance in the qualitative analysis of mixed bodies, that every reagent which is found to produce a precipitate be applied in sufficient quantity to throw down completely the whole of the substance it is capable of precipitating, otherwise the action of the remaining reagents will be materially interfered with.

DETERMINATION OF BASES.

Pass sulphuretted hydrogen gas through the solution made slightly acid.

I. EXAMINATION OF THE PRECIPITATE BY SULPHURETTED HYDROGEN.

This precipitate may contain the sulphurets of the metals Class I. page 57., sulphur from decomposition of sulphuretted hydrogen by perox. of iron and chromic acid, and sulphurets of arsenic from arsenious or arsenic acid.

Collect on a filter, and wash quite clean with distilled water; remove as much of the precipitate as possible from the filter, and treat it first with a little ammonia, and then with hydrosulph. of ammonia.

Sulphurets undissolved :—Cadmium, lead, bismuth, copper, silver, subsulphuret and protosulphuret of mercury. Collect on a filter and wash. Examine a portion for *cadmium* by carb. soda on charcoal before the blowpipe. (See Blowpipe Tables.) Digest the remainder in strong nitric acid, and apply heat. Sulphur separates. (Sulphate of lead may be formed.)

Sulphurets undissolved : protosulph. and subsulph. of *mercury*.

If *protosulphuret*, it may be sublimed in a tube without change.

If *subsulphuret*, it gives on sublimation a mixture of metallic mercury and protosulphuret.

Solution may contain oxides of cadmium, bismuth, lead, copper, and silver. (Cadmium has been previously sought by the blowpipe.) Divide the solution into two portions: test one with ammonia in ex. for *copper*. Evaporate off the excess of acid of the other portion, and dilute with water.

If a precip. on dilution, it is from *bismuth*. Filter, and add sulphate of soda to the solution.

If a white precipitate, this is sulphate of *lead*. Filter : solution may contain *silver*; chloride of sodium gives a white curdy precipitate.

Sulphurets dissolved :—Gold, antimony, protosulph. and bisulph. of tin. (Arsenic.) Add hydrochloric acid to the solution, and apply heat; filter, evaporate, and pour into water; a bulky white precipitate indicates *antimony*.

Test the original solution with protochloride of tin for *gold* (gives purple of Cassius), and with chloride of gold for protox. or protochloride of *tin*. If no protox. of tin, examine for *peroxide*, by reducing to the metallic state with carb. soda on charcoal. (See Blowpipe Tables.)

II. TREATMENT OF THE SOLUTION FILTERED FROM THE PRECIPITATE BY SULPHURETTED HYDROGEN.

Render it slightly alkaline by ammonia, and then add a very slight excess of hydrosulphate of ammonia. The precipitate may consist of alumina, oxide of chromium, sulphurets of nickel, cobalt, iron, manganese, and zinc, and if the substance for analysis be insoluble in water, magnesia, lime, barytes, and strontian united with phosphoric, arsenic, and boracic acids, or their metallic bases with fluorine.

Filter; wash the precipitate with water to which a little ammonia has been added, and digest it in pure dilute hydrochloric acid, until the odour of sulphuretted hydrogen is no longer perceptible.

Sulphurets of *nickel* and *cobalt* remain undissolved; filter and test for these by microcosmic salt or borax before the blowpipe. (See Blowpipe Tables.)

TREATMENT OF THE SOLUTION IN HYDROCHLORIC ACID OF THE PRECIPITATE BY HYDROSULPHATE OF AMMONIA.

Add first a little nitric acid to peroxidize iron, then muriate of ammonia, and afterwards excess of caustic ammonia.

(a) If the substance is *soluble in water* (or a metallic alloy), the precipitate may contain alumina, oxide of iron, and oxide of chromium. Redissolve in hydrochloric acid, and boil with excess of caustic potash.

Precipitated—oxide of *chromium* and oxide of *iron*; test by blowpipe for chromium. Redissolve in hydrochloric acid, and test with yellow pruss. potash for iron.

(b) If the substance is insoluble in water, the precipitate may contain, besides the bodies in (a), phosphates or borates of magnesia, barytes, lime, and strontian. Dissolve in a little hydrochloric acid and add dilute sulphuric acid to a portion.

Precipitated — *barytes*, *strontian*, and perhaps *lime*: filter. If a precipitate, neutralize another portion of the hydrochloric acid solution, and apply the following tests:—

1. Hyposulphite of soda precipitates barytes, but not strontian.
2. Solution of sulphate of lime precipitates barytes, but not strontian.
3. Hydrofluosilicic acid precipitates barytes, but not strontian.

Solution may contain zinc manganese, and traces of nickel and cobalt: filter. Add excess of caustic potash.

Precipitated—*nickel*, apple green; *manganese*, brownish, becoming darker on exposure. (Test for manganese by borax, before the blowpipe.) In solution — *zinc* and *cobalt*: filter. Add hydrosulph. of ammonia, and examine precipitate by blowpipe tests for these metals. (For cobalt, borax; for zinc, reduction with soda on charcoal.)

Alumina is dissolved; precipitated by muriate of ammonia.

1. Examine a portion of the precipitate for oxide of *chromium* by borax before the blowpipe.
2. Dissolve the remainder in hydrochloric acid, and test one portion by yellow pruss. potash for *iron*.
3. Neutralize the other portion by ammonia, and test for *magnesia* by phosphate of soda.

III. EXAMINATION OF THE SOLUTION FILTERED FROM THE PRECIPITATE BY HYDROSULPHATE OF AMMONIA.

This may contain barytes, strontian, lime, magnesia, potash, soda, and lithia.

Add hydrochloric acid in slight excess, boil to expel sulphuretted hydrogen, and filter. Supersaturate the filtered solution with carbonate of ammonia.

Precipitated—lime, barytes, strontian. Dissolve in hydrochloric acid.

1. Precipitate a portion of the solution by dilute sulphuric acid, and filter:—if the filtered solution (nearly neutralized with ammonia) give a precipitate with oxalic acid, this is oxalate of lime.
2. To another portion add a solution of sulphate of lime:—if a precipitate, this is sulphate of barytes.
3. The solution evaporated to dryness communicates a strong crimson colour to the flame of alcohol, if it contain strontian.

In solution, magnesia, potash, soda, and lithia.

Evaporate to dryness, and ignite at a red heat to expel ammoniacal salts. Redissolve in water, and add a saturated solution of sulphuret of barium.

Magnesia is precipitated.

Add sulphuric acid to the solution, filter to separate sulphate of barytes, evaporate to dryness, and ignite. Redissolve in water.

1. To one portion of the solution add phosphate of soda and ammonia:—a white precipitate, after some time, indicates lithia. (Salts of lithia communicate a crimson colour to flame.)
2. In another portion test for potash by an excess of tartaric acid.
3. Salts of soda communicate a strong yellow colour to flame.

[For the method of detecting alkalies in bodies insoluble in acids, see Note, p. 63.]

DETERMINATION OF ACIDS.

Effervescence on adding hydrochloric acid may proceed from carbonic, sulphurous, and hydrosulphuric acids: the last is detected by its odour and action on salts of lead; for detection of the two first see below.

Make the solution neutral, and add a solution of nitrate of barytes.

Precipitated — sulphuric, arsenic, iodic, boracic, hydrofluoric, phosphoric, and if no acid has been applied, carbonic and sulphurous.

Filter, and add hydrochloric acid. Effervescence may occur of sulphurous and carbonic acids. To detect *sulphurous* apply the test of iodic acid and starch. If not sulphurous, it is *carbonic* acid. Transmit the gas through lime-water. [Peroxide of lead separates carbonic and sulphurous acids by absorbing the latter.]

Insoluble in hydrochloric acid, *sulphuric* (sulphate of barytes).

Filter, and pass sulphuretted hydrogen gas through the solution in hydrochloric acid, diluted.

Precipitated: —

In solution, *phosphoric, boracic, and hydrofluoric*. Apply the tests mentioned in Table (B.), p. 64.

Sulphuret of arsenic from *arsenic* or *arsenious* acid (reduce by black flux).

Iodine from *iodic* acid (apply sulphurous acid and starch to a portion of the original solution).

In solution — hydrochloric, hydriodic, hydrosulphuric (as a soluble sulphuret), nitric and chloric. Add a solution of nitrate of silver.

Add excess of ammonia to the precipitate; filter.

If the insoluble portion is black, it contains a *sulphuret*; if yellow, an *iodide*. Solution contains *chloride* of silver: nitric acid in excess gives a white precipitate.

In solution, *nitric and chloric*; filter. For these apply the tests mentioned in Table (C.), p. 65.

To a portion of the original solution add a salt of lead or mercury. A black sulphuret is first precipitated, afterwards a yellow or red iodide. (See special tests for hydriodic and hydrosulphuric acids).

SECTION III.

QUALITATIVE ANALYSIS OF A SUBSTANCE WHICH MAY CONTAIN ALL INORGANIC BODIES WHOSE PROPERTIES ARE WELL ASCERTAINED.

The solution effected either in water, acids, or by fusion with an alkali, and subsequent solution in an acid.

I. EXAMINATION FOR BASES AND SOME ACIDS.

Acidify the solution, if neutral, by hydrochloric or nitric acid. (See Table, p. 60., for cases in which hydrochloric acid must not be used.)

Substances precipitated by hydrochloric or nitric acid aided by heat, *insoluble in an excess*: — silicic acid; tungstic acid; tantalic acid; titanic acid: — by strong nitric acid only; — peroxide of tin. These are distinguished from each other by blowpipe tests. (See Tables on the Blowpipe.)

If a precipitate occur, filter; transmit sulphuretted hydrogen gas through the solution to saturation, set aside for twelve hours, then boil to expel excess of sulphuretted hydrogen, and filter.

TREATMENT OF THE PRECIPITATE FORMED BY SULPHURETTED HYDROGEN.

Wash the precipitate with sulphuretted hydrogen water, add to it a little ammonia, and digest in hydrosulphate of ammonia : filter.

Sulphurets undissolved : — cadmium, bismuth, lead, silver, copper, mercury, osmium, palladium, rhodium.

To detect the first six metals, proceed according to Table, page 66.

After the application of sulphate of soda to detect lead, and hydrochloric acid for silver, neutralize the solution by carbonate of soda, and add cyanide of mercury ; if a white gelatinous precipitate falls, this is cyanide of *palladium*.

Osmium is ascertained by the fumes of osmic acid evolved when the substance is boiled with nitric acid.

To discover *rhodium*, reduce to the metallic state by heating the substance in hydrogen gas ; metallic rhodium is insoluble in nitric acid and aqua regia, but dissolves by fusion with bisulphate of potash.

Sulphurets dissolved : — tin, gold, antimony, arsenic, platinum, iridium, molybdenum, tellurium, selenium.

Examine for *tin*, *gold*, and *antimony*, according to Table, page 66.

Precipitate the solution in hydrosulph. ammonia by hydrochloric acid in slight excess ; examine a portion of the precipitate before the blowpipe for *molybdenum*, *arsenic*, and *selenium* ; dissolve the remainder in aqua regia ; evaporate off excess of acid, and dilute. If the absence of selenium has been proved by the blowpipe, and of gold by protochloride of tin, the production of a black precipitate on adding sulphurous acid to this solution indicates *tellurium*. If selenium and gold also exist, tellurium must then be sought by the blowpipe. Add a concentrated solution of chloride of potassium.

Precipitated — chloride of platinum and potassium, and chloride of iridium and potassium. Filter ; ignite with an equal weight of carbonate of potash, wash with water, and treat with hot aqua regia. The solution may be precipitated by bicarbonate of potash, and the precipitate examined for tin and antimony by the blowpipe.

Peroxide of *iridium* is undissolved. Solution contains *platinum* ; chloride of potassium gives a yellow precipitate.

TREATMENT OF THE SOLUTION FILTERED FROM THE PRECIPITATE BY SULPHURETTED HYDROGEN.

Add first ammonia, and then a slight excess of hydrosulph. of ammonia: expose to the air until the excess of hydrosulph. ammonia is dissipated; filter, if a precipitate be formed, and wash with very dilute ammonia.

Precipitated — sulphurets of zinc, iron, manganese, nickel, cobalt, and uranium; alumina, glucina, thorina, yttria, zirconia, oxide of cerium, oxide of chromium; and if the substance under examination is insoluble in water, certain salts of barytes, strontian, lime, and magnesia. When well washed, dissolve in aqua regia, evaporate off excess of acid, dilute, and add sulphuric acid mixed with a little alcohol.

In solution — *magnesia, barytes, lime, strontian, potash, soda, lithia*. To distinguish these, see Table, page 69.

Precipitated — sulphates of *barytes, lime, and strontian*. See Table, page 68. Filter from the precipitate by sulphuric acid, add muriate of ammonia, and excess of caustic ammonia.

Digest the precipitate by ammonia in cold caustic potash.

In solution — *zinc, manganese, nickel, cobalt*. See Table, page 68.

Undissolved by potash — perox. of iron, zirconia, thorina, ox. of cerium, yttria, perox. of uranium, phosphate of magnesia. Filter, wash, dissolve in hydrochloric acid, and add a large excess of bicarbonate of potash.

In solution — alumina, glucina, oxide of chromium; add carbonate of ammonia in excess.

Undissolved — perox. iron; (dissolve in hydrochloric acid and test with pruss. of potash;) *phosphate of magnesia; phosphate of yttria.* (?)

Precipitated — alumina and oxide of chromium; boil with a strong solution of carbonate of potash.

In solution — *glucina*; neutralize with hydrochloric acid, and precipitate by ammonia. (A trace of ox. of chromium.)

Zirconia is precipitated. Neutralize the filtered liquid, and add excess of a hot saturated solution of sulphate of potash, boil and dilute slightly.

Alumina, undissolved; confirm by blowpipe, with nitrate of cobalt. Oxide of *chromium* is dissolved; confirm by the blowpipe.

Precipitated — *thorina* and *ox. of cerium*; the latter is detected by the blowpipe.

In solution — *perox. uranium* and *yttria*; ammonia gives a yellow precipitate of perox. uranium. Confirm by blowpipe.

II. EXTENDED EXAMINATION FOR ACIDS.

Acids to be sought besides the list, page 58.

Bromic	Hyposulphurous	Phosphorous
Chromic	Hyposulphuric	Selenic
Hydrobromic (bromides)	Manganic	Selenious
Hypophosphorous	Nitrous	Silicic.
Hypermanganic	Oxalic	

The acids already discovered in the determination of the bases are — arsenic, chromic, manganic, and selenious or selenic.

The evolution of chlorine, when the substance is digested in hydrochloric acid, may proceed from chloric, iodic, bromic, selenic, manganic, and hypermanganic acids.

The first operation consists in mixing the substance, either in the dry state or in a strong solution with concentrated sulphuric acid, and applying heat in a test tube. Observe whether any volatile acid is evolved by holding a rod moistened with ammonia over the tube.

Sulphurous acid evolved, known by its odour and action on iodic acid.	Sulphurous.	{ Leaves a clear solution; soluble sulphites dissolve sulphur.
	Hyposulphurous.	{ Leaves a milky solution from precipitation of sulphur: hyposulphites dissolve moist chloride of silver.
	Hyposulphuric.	{ Leaves sulphuric acid; if pure hydrochloric acid be used instead of sulphuric, the liberated sulphuric acid may be observed.
Coloured gases or vapours evolved.	Bromates and bromides give the yellowish red vapour of bromine.	{ Oxygen gas is evolved from bromates by the action of sulphuric acid. Bromates part with oxygen when heated to redness.
		{ From bromides, chlorine water liberates bromine, giving a yellow solution, from which the bromine is removed by agitation with ether.
	Nitrous.	{ Nitric oxide is evolved and forms ruddy fumes with the air. Nitric acid remains.
	Chloric.	{ Peroxide of chlorine liberated; the liquid bleaches vegetable colours.
	Hydriodic (iodides).	{ Fumes of iodine. Apply the test of chlorine water and starch to the solution of the substance.

Effervescence of carbonic acid.	Carbonic.	Effervescence in cold with dilute acid. No effervescence in cold. Chloride of calcium gives a white precipitate, which effervesces with hydrochloric acid after, but not before ignition.
	Oxalic.	

Note. — If carbonic acid, a jar of the gas collected over water, agitated with a little lime-water, redissolves the precipitate it produces at first. If it be mixed with sulphurous acid gas, the last may be separated by its greater solubility in water, or by agitation with peroxide of lead.

The other acids volatilized when the substance is heated with sulphuric acid, are — hydrosulphuric, hydrofluoric, hydrochloric, and nitric. (Examine for these according to the Tables (A.), (B.), and (C.), pp. 64, 65.)

Acids not volatilized when their combinations are heated with sulphuric acid, are — arsenic, iodic, phosphoric, boracic, sulphuric, silicic, selenious, selenic, hypophosphorous, and phosphorous acids. (For the first five apply the special tests mentioned in Table (B.), p. 64.)

Silicic.	Acidulate the solution with hydrochloric acid, evaporate to dryness, and redissolve in dilute acid. Silicic acid remains undissolved. Confirm by blowpipe with carbonate of soda.
Selenic and selenious acids.	Detected by the blowpipe. These acids are distinguished from each other by sulphuretted hydrogen, which precipitates sulphuret of selenium from selenious, but not from selenic acid. By boiling with hydrochloric acid, selenic is reduced to selenious acid, and is then precipitated by sulph. hydrogen.
Phosphorous and hypophosphorous acids.	These acids reduce salts of mercury and gold to the metallic state in acid solutions. Most phosphites and hypophosphites evolve phosphuretted hydrogen when heated to redness, the gas sometimes being spontaneously inflammable.

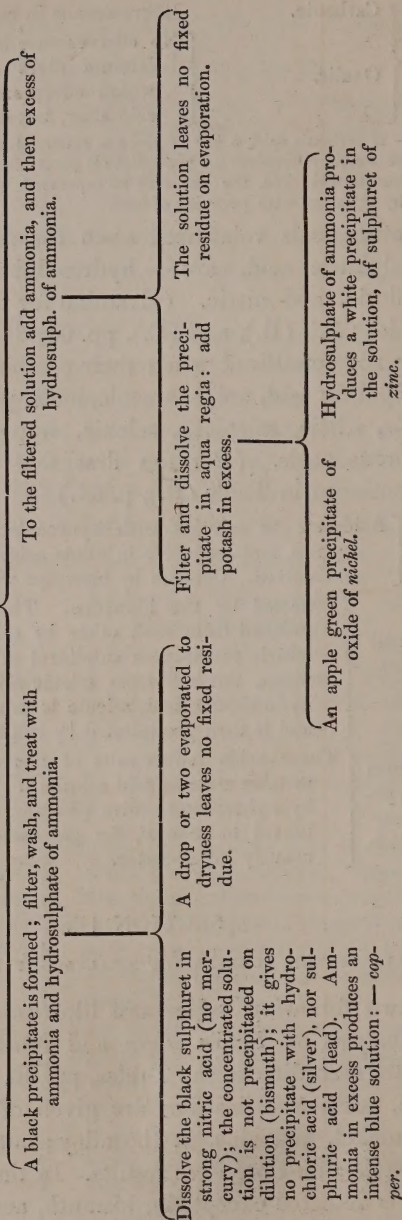
SECTION IV.

EXAMPLES OF THE QUALITATIVE ANALYSIS OF METALLIC ALLOYS.

The two following tables are illustrations of the qualitative analysis of *Berlin silver* and *Newton's fusible metal* (conducted according to the Tables, pp. 66. 68.). In the first example, positive indications are given of the existence of copper, nickel, and zinc, in the alloy; but the tests for all other metals afford negative results. In the second example, nothing is detected except tin, bismuth, and lead.

I. BERLIN SILVER.

Dissolve the metal in dilute nitric acid, evaporate off most of the excess of acid, and dilute with water.
Transmit sulphuretted hydrogen gas through the solution, and filter.



The constituents of Berlin silver are therefore copper, nickel, and zinc.

II. NEWTON'S FUSIBLE METAL.

Dissolve in dilute nitric acid or aqua regia, with a gentle heat; if a white precipitate be formed, this must be examined before the blowpipe for *tin* (as below). Dilute the acid solution; transmit sulphuretted hydrogen, and filter.

A precipitate falls, which is collected on a filter, and treated with ammonia and hydrosulphate of ammonia. The solution leaves *no* fixed residue on evaporation.

Wash the insoluble portion, and digest in strong nitric acid. No black sulphuret remains undissolved, hence, mercury is absent. The excess of acid being evaporated from the solution in nitric acid, on pouring the liquid into water a white precipitate appears, arising from the presence of *bismuth*. Filter, and add a solution of sulphate of soda.

A few drops of the solution leave a fixed residue when evaporated. Neutralize the liquid with hydrochloric acid; a precipitate falls, which, heated with soda on charcoal before the blowpipe, gives a button of metallic *tin*, recognised by its ready fusibility and malleability.

A white precipitate appears, which is sulphate of *lead*. The filtered solution gives no precipitate with hydrochloric acid; hence, no silver is present.

Newton's fusible metal, therefore, contains bismuth, lead, and tin.

SECTION V.

QUALITATIVE ANALYSIS OF SILICATES.

Means of effecting solution. — The first operation consists in the reduction of the mineral to an impalpable powder, its perfect solution depending greatly on the minuteness of its division. It is first crushed between folds of paper in a vice, or on an anvil with a hammer; or if the mineral is too hard for this, in a steel diamond mortar, the powder being in this case digested in dilute hydrochloric acid * to remove any portions of iron it may have acquired from the mortar. Its pulverization is completed in an agate mortar, and, if necessary, by levigation, affusion of water, and decantation. When the mineral is extremely hard, it is apt, during pulverization, to scratch the mortar, and thus acquire some portions of agate which have been rubbed off. The amount of agate (which is nearly pure silica) thus acquired by the powder must always be ascertained in accurate quantitative analyses, otherwise a serious error is involved. It is necessary to weigh the mortar before and after pulverization, and to deduct the quantity of silica lost from the weight of the mineral employed, and also from that of the silica obtained in the analysis.

A portion of the powdered mineral is next treated with concentrated hydrochloric acid, in a porcelain bason, with the application of a gentle heat. Some silicates are immediately acted on by the acid; others only by a very long digestion; and some, in fact the greater number, in no degree whatever.

If fully decomposed by hydrochloric acid, all the bases formerly united to silica have become chlorides, and are dissolved on the addition of a little water, while the silica remains in the free state, generally in the form of a light flocculent jelly. If any gritty particles are perceived on

* The harder silicates are not acted on by hydrochloric acid.

stirring with a glass rod against the bottom of the bason, the mineral is not entirely decomposed.

Silicates which resist the action of hydrochloric acid may be rendered soluble in it by first fusing them (in the most minute state of division) with three or four times their weight of carbonate of potash or soda; a silicate of the alkali is then formed, carbonic acid gas being at the same time evolved. The two powders may be mixed in the platinum crucible in which the fusion is to be performed, by means of a glass rod; the crucible is covered, brought gradually to a heat sufficiently high to fuse the mass, and sustained at this temperature for half an hour. The fused mass is then moistened with water, removed from the crucible into a porcelain bason, and treated with pure hydrochloric acid. Effervescence of carbonic acid now occurs; the bases formerly united to silica dissolve in the acid, becoming chlorides, and silica remains in the state of an insoluble jelly.

To avoid loss of solutions by effervescence, it is necessary, in quantitative analyses, to place the capsule containing the solution within a larger one, to retain the projected drops, which must afterwards be washed into the solution; or a funnel, whose diameter at top is a little less than that of the bason, may be inverted within it, so that the drops spirted within the funnel shall fall again into the bason.

There are a few silicates which resist the action of even carbonates of soda and potash; to effect the decomposition of these, caustic potash, to the amount of about four or five times the weight of the mineral, must be employed instead of the alkaline carbonate, and the heating then performed in a silver instead of a platinum crucible, heated carefully over the spirit lamp with a circular wick. The crucible must be well closed with its lid, as the escape of water from the hydrate of potash produces violent ebullition, which might cause a serious loss in quantitative analyses. A mixture of one part of hydrate of potash and three of carbonate of soda is found convenient for this operation, as the fusion may then be performed in a platinum crucible.

When the mineral is to be examined for alkalies, it is

obvious that neither carbonate of potash nor of soda can be used: instead of these, if insoluble in hydrochloric acid, having made a previous investigation of the other constituents by the ordinary methods, the mineral is decomposed by heating with carbonate of barytes, or, what is preferable, quick-lime; the last, however, requires a very intense heat. The solution in hydrochloric acid is made in the same manner as if an alkaline carbonate had been employed: barytes is separated from the solution by sulphuric acid; or if lime be used, this earth is removed by a solution of carbonate of ammonia.

The ordinary constituents of silicates are, silicic acid, alumina, magnesia, lime, protoxide of iron, oxide of manganese, potash, and soda. The following tables contain directions for the detection of each of these, but it rarely happens that more than three or four bases exist in the same mineral.

The only known natural silicates which contain other ingredients, as essential constituents, than those which may be discriminated by the tables, are the following: —

Names of the particular Constituents.	Names of the Minerals.
Barytes - - -	Barytes-harmotome; brewsterite.
Boracic acid - -	{ Axinite; botryolite; datholite; tourmaline; mica; lepidolite; pinit.
Carbon - - -	{ Pyrorthite. (Many minerals contain a little organic matter.)
Cerium, oxide of -	{ Cerite; cerine; gadolinite; orthite; pyror- thite.
Chlorine - - -	Sodalite; eudialite; pyrosmalite.
Chromium, oxide of -	{ Several minerals having a green colour, as emerald, serpentine, &c.; pyrope.
Copper, oxide of -	{ Diopase; chrysocolla; cyprine; cerine, allo- phane.
Fluorine - - -	{ Apophyllite; chondrodite; chabasite; horn- blende; mica; carpholite; scapolite; topaz.
Glucina - - -	{ Cymophane; euclase; gadolinite; helvine; beryl.

Names of the particular Constituents.	Names of the Minerals.
Lead, oxide of -	- Thorite ; siliceous oxide of zinc.
Nickel, oxide of -	- Chrysoprase ; pimelite ; olivine.
Phosphoric acid -	- Lepidolite ; sordawalite.
Strontian - -	- Brewsterite.
Sulphur - -	- Helvine ; haüyne ; spinellane ; lapis-lazuli.
Tantallic acid -	- Traces in emerald.
Thorina - -	- Thorite.
Tin, oxide of -	- { Traces in emerald ; euclase ; siliceous oxide of zinc ; thorite ; olivine.
Uranium, oxide of -	- Pitchblende ; thorite.
Ytria - -	- Gadolinite ; orthite ; pyrrorthite.
Zinc, oxide of -	- Siliceous oxide of zinc.
Zirconia - -	- Zircon ; eudialite.

QUALITATIVE ANALYSIS OF SILICATES.

Solution made either in hydrochloric acid, or by fusion with an alkali and solution in hydrochloric acid. Evaporate to dryness, and treat again with dilute hydrochloric acid; the residue undissolved is *silicic acid*. If protoxide of iron is present (ascertained by red prussiate of potash), peroxidize it by heating with a little nitric acid.

To the solution add muriate of ammonia and excess of caustic ammonia.

Precipitate may contain alumina and peroxide of iron: filter and wash. Redissolve in a little hydrochloric acid, add an excess of potash, and boil.

Oxide of iron remains undissolved by potash. *Alumina* is redissolved. (Muriate of ammonia reprecipitates alumina.)

The filtered solution may contain magnesia, oxide of manganese, lime, potash, and soda: evaporate off most of the excess of ammonia, add oxalate of ammonia, boil and filter.

Precipitate is oxalate of lime. (It may also contain a trace of oxalate of manganese.)

To the filtered solution add hydrosulphate of ammonia.

Precipitate is sulphuret of *magnesia*. Confirm by blowpipe.

Destroy the excess of hydrosulph. ammonia by evaporation and decomposition by hydrochloric acid. To a portion of the solution add ammonia (if acid) and phosphate of soda. If a white precipitate is formed on agitation, this is phosphate of soda.

If no magnesia is present, and no fixed alkali has been added, evaporate the other portion of the solution to dryness and ignite. Examine the residue by the tests for *potash* and *soda*. (p. 63.) If magnesia is present, and no fixed alkali has been added, after precipitation by hydrosulph. ammonia and filtration, evaporate to dryness, ignite, and redissolve in water. The solution may contain chloride of potassium, chloride of sodium and chloride of magnesium. Add a strong solution of caustic barytes.

Magnesia is precipitated.

Add sulphuric acid to the solution.

Sulphate of barytes is precipitated. Evaporate the solution to dryness, ignite the residue, and redissolve in water. Examine for *potash* and *soda*.

(p. 63.)

ANALYSIS OF A SILICATE INSOLUBLE IN HYDROCHLORIC ACID, SUSPECTED TO CONTAIN AN ALKALI.

Calcine the mineral with five or six times its weight of carbonate of barytes, and treat the ignited mass with hydrochloric acid. Silica remains undissolved. To the solution in hydrochloric acid add carbonate of ammonia and hydrosulphate of ammonia.

Precipitate may contain carbonate of barytes, carbonate of lime, alumina, sulphuret of manganese and sulphuret of iron. Filter and wash.

Solution may contain magnesia, alkali, traces of lime, and barytes. Add a solution of oxalate of ammonia.

Lime and barytes are precipitated.

Evaporate the solution to dryness, ignite the residue, redissolve in water, and add a strong solution of caustic barytes.

Magnesia is precipitated.

The solution contains alkali and barytes. Add dilute sulphuric acid.

Sulphate of barytes is precipitated. Evaporate the filtered solution to dryness, ignite and redissolve in water. Examine for *potash* and *soda*. (Table, page 63.)

EXAMPLES OF THE QUALITATIVE ANALYSIS OF SILICATES.

1. STILBITE.

Dissolve the powdered mineral in hydrochloric acid; evaporate to dryness, and treat the residue with dilute hydrochloric acid.

Insoluble residue is *silica*. (Red prussiate of potash gives no blue precipitate.) Add excess of caustic ammonia. (As free hydrochloric acid is present, the addition of muriate of ammonia is unnecessary.)

A white precipitate falls, which is entirely soluble in potash; hence this is *alumina*.

Add oxalate of ammonia to the solution.

Oxalate of *lime* is precipitated.

A drop of the solution evaporated to dryness on platinum foil leaves no fixed residue after ignition.

The constituents of stilbite are therefore silica, alumina, and lime.

2. HORNBLENDE.

After fusion with carbonate of soda, dissolve in hydrochloric acid, and separate *silica* by evaporation and re-solution of the residue. After a little nitric acid has been added to peroxidize iron, add, first muriate of ammonia (unless the solution is very acid), and then caustic ammonia.

The precipitate is filtered and dissolved in hydrochloric acid. To the acid solution add excess of potash, and boil.

Add oxalate of ammonia to the filtered solution.

Peroxide of *iron* remains undissolved.

Muriate of ammonia added to the alkaline solution produces a precipitate of *alumina*.

Oxalate of *lime* is precipitated.

Phosphate of soda gives a white precipitate of phosphate of *magnesia* and ammonia.

Hornblende therefore contains silica, alumina, oxide of iron, lime, and magnesia.

3. LEPIDOLITE.

(1.) Dissolve in hydrochloric acid, after fusion with carbonate of soda. *Silica* is separated in the usual manner.

Add muriate of ammonia and caustic ammonia to the solution.

A white precipitate, almost wholly soluble in potash; hence *alumina*. (Sometimes a trace of manganese remains.)

Oxalate of ammonia produces no precipitate in the filtered solution. Hydrosulphate of ammonia gives a flesh-colour precipitate of sulphuret of *manganese*.

(2.) For alkalis by fusion with carbonate of barytes.

To the solution in hydrochloric acid separated from silica (page 83.) add carbonate of ammonia and hydrosulphate of ammonia.

Precipitate contains carbonate of barytes, carbonate of lime, alumina, and sulphuret of manganese: filter.	Add a solution of oxalate of ammonia to the filtered solution.	
	Traces of barytes and lime are precipitated.	The solution leaves a residue on evaporation and ignition. Tartaric acid and chloride of platinum indicate the presence of <i>potash</i> . Phosphate of soda and ammonia gives a white precipitate, on long standing, of phosphate of lithia and soda. Lithia is also discoverable by the blowpipe.

Lepidolite therefore contains silica, alumina, manganese, potash, and lithia.

SECTION VI.

QUALITATIVE ANALYSIS OF MINERAL WATERS.*

The first point to be determined in the examination of a mineral water is, to which of the five great classes of waters does the one in question belong?

1. *Carbonated*, — containing free carbonic acid.
2. *Sulphureous*, — containing sulphuretted hydrogen.
3. *Chalybeate*, — containing carbonate of iron.
4. *Alkaline*, — containing carbonate of soda.
5. *Saline*, — containing much salts.

1. If the water reddens blue litmus paper before boiling, but not afterwards, and the blue colour of the reddened paper is restored on warming, it is carbonated.

2. If it possesses a nauseous odour, and gives a black precipitate with acetate of lead, it is sulphureous.

3. If after the addition of a few drops of hydrochloric acid it gives a blue precipitate with yellow or red prussiate of potash, the water is a chalybeate.

* If practicable, the water should always be collected near the mouth of the spring, and secured in well closed vessels.

4. If it restores the blue colour to reddened litmus paper, after boiling, it is alkaline.

5. If it possesses neither of the above properties in a marked degree, and leaves a large residue on evaporation, it is a saline water.

The substances which commonly enter into the composition of a mineral water are, —

Acids. — Sulphuric, carbonic, phosphoric, silicic, hydrochloric (chlorides).

Bases. — Potash, soda, lime, oxide of iron, magnesia, alumina.

Besides these, other constituents are sometimes found, but they are comparatively of much rarer occurrence: these are,—

Acids. — Nitric, sulphurous, boracic, and some organic acids produced by the decomposition of vegetable matter (crenic, apocrenic, and puteanic).

Bases. — Lithia, strontian, oxide of manganese, oxide of zinc, oxide of copper.

To determine with accuracy on all the ingredients which may exist, the water should be concentrated by evaporation nearly to dryness, although a cursory examination, sufficient for many purposes, may be performed on the water in its ordinary state. On boiling, most waters evolve a gas, which in carbonated, alkaline, and chalybeate waters is chiefly carbonic acid; and in sulphureous, sulphuretted hydrogen: all waters which are exposed to the atmosphere at ordinary temperatures contain also oxygen and nitrogen, though not in the proportions in which these gases exist in the atmosphere. In an extended examination the liberated gas must be collected in the mercurial trough.*

While the gas is evolved by ebullition, a precipitate is formed in chalybeate, carbonated, and most saline waters, which may contain silica, carbonates of lime, magnesia, and strontian, peroxide of iron, fluoride of calcium, and phosphate of alumina. As this precipitate is to be collected and analyzed, particular attention must be paid to the perfect filtration of the water before evaporation, that no solid matter

* For means of collecting the gas, see the "Quantitative Analysis of Mineral Waters."

diffused through the water may contaminate the precipitate actually deposited from a state of solution.

When the water is evaporated to dryness, and the residue treated with distilled water, the precipitate formed during ebullition remains undissolved, while all the soluble matters dissolve, forming a solution which may contain potassium, sodium, calcium, and magnesium, combined with chlorine, iodine, and bromine, or as nitrates or sulphates of the oxides of these metals.

By evaporating to dryness, and treating the residue with water, three classes of constituents therefore become separated, each of which is to be examined independently. This is the first step in the analysis: 1st, the gases held in solution in the water in its natural state; 2d, the bodies held in solution by the gas, and rendered insoluble by evaporation; and 3d, bodies which are permanently soluble, and therefore redissolved when the dry residue is treated with pure water. The qualitative analysis of each class is conducted in the following manner:—

I. FOR THE GASES EVOLVED.

Into the gas contained in a jar over mercury introduce a little solution of potash free from carbonate.

Absorbed — carbonic acid and sulphuretted hydrogen. Test the original water with lime-water for *carbonic* acid, and with acetate of lead for *sulphuretted hydrogen*. (pp. 89, 90.)

Not absorbed by potash — oxygen and nitrogen: introduce a stick of phosphorus.

Oxygen is absorbed.

Nitrogen remains.

II. FOR THE BODIES RENDERED INSOLUBLE BY EVAPORATION.

Treat the matter with pure hydrochloric acid, and evaporate to dryness. Reserve a small portion of the residue to test for *fluorine*, by heating with sulphuric acid in a glass tube, and treat the remainder with dilute hydrochloric acid. The insoluble residue is *silica*.

EXAMINATION OF THE SOLUTION IN HYDROCHLORIC ACID OF THE PRECIPITATE FORMED ON EBULLITION.

The second solution in hydrochloric acid may contain lime, magnesia, strontian, lithia, phosphate of alumina, oxide of iron (potash and soda). (Liebig, *Handwörterbuch*.)

Add excess of ammonia.

The solution contains lime, magnesia, strontian, lithia, manganese; decompose by hydrosulph. of ammonia.

Saturate the solution with a free acid, and boil; add oxalate of ammonia.

The solution contains magnesia, lithia, (soda, potash); evaporate to dryness, calcine, and dissolve in water.

To the solution add sulphuret of barium; decompose by sulphuric acid; filter, evaporate, and calcine. The residue consists of sulphates of the alkalis.

The insoluble residue, together with the precipitate produced in the solution by sulphuret of barium, contains all the *magnesia*. This earth is separated from barytes by sulphuric acid.

Sulphuret of *manganese* is precipitated.

The precipitate is oxalate of lime and oxalate of strontian; convert into dry and neutral nitrates, and treat with alcohol.

The solution contains nitrate of *lime*.

The insoluble residue is nitrate of *strontian*.

The precipitate may contain oxide of iron, phosphate of alumina, traces of magnesia and manganese. Filter and dissolve in hydrochloric acid; add excess of potash, and boil.

The solution contains *alumina*, phosphoric acid. Supersaturate with an acid, and then add excess of ammonia. *Alumina*, or *phosphate of alumina*, is precipitated.

The precipitate contains oxide of iron (traces of manganese), phosphoric acid, magnesia. Treat with muriate of ammonia, and add hydrosulph. of ammonia.

In solution are traces of *phosphoric acid* and *magnesia*.

The precipitate consists of sulphuret of *iron*, with a trace of *manganese*.

III. EXAMINATION OF THE BASES IN THE SOLUTION FILTERED FROM THE PRECIPITATE PRODUCED BY EBULLITION.

This may contain lime, magnesia, potash, soda (*lithia*).

If lime is present, oxalate of ammonia gives a white precipitate of oxalate of *lime*. Filter the solution from the precipitated oxalate, and add phosphate of ammonia and a little free ammonia to test for magnesia, which should produce a white precipitate of phosphate of *magnesia* and ammonia, if magnesia is present.

To another portion of the water add a solution of caustic barytes.

The precipitate is magnesia and lime (sulphate of barytes).	The filtered solution contains lime, barytes, and alkalies : add carbonate of ammonia.	
	Precipitate is carbonate of lime and carbonate of barytes.	The solution contains ammoniacal salts, <i>potash, soda (lithia)</i> . Evaporate, and calcine to expel ammoniacal salts; the residue consists of alkaline chlorides. Dissolve in water, and examine by chloride of platinum, tartaric acid, and blowpipe.

To discover *lithia*, a large quantity of the water should be evaporated to dryness, the residue dissolved in a small quantity of water, and filtered to separate the insoluble portion. To the solution, phosphate and carbonate of soda are added, the mixture is evaporated to dryness, and the residue treated with water, when, if *lithia* is present, this alkali remains undissolved, in the state of phosphate of *lithia* and soda.

IV. EXAMINATION FOR ACIDS.

First Portion. *Carbonic acid* and *sulphuretted hydrogen*.

—If the water, fresh from its source, feebly reddens blue litmus paper, and the colour of the paper is restored on gently heating it, this indicates the presence of *carbonic acid*. Lime-water then occasions a precipitation of carbonate of lime.

Carbonic acid may exist in three different states in mineral waters. 1st, as the free uncombined acid dissolved in the

water; 2d, holding in solution earthy carbonates; and 3d, combined with potash or soda as bicarbonates. These different states may be distinguished by the following characters: — 1st. If the precipitate of carbonate of lime produced in the water by the addition of a small quantity of lime-water is redissolved by an excess of the mineral water, it contains the uncombined acid. 2d. If, on the contrary, the precipitated carbonate of lime does not dissolve in any excess of the water, and during ebullition a precipitate is formed which effervesces with acids, the carbonic acid is combined with earthy carbonates. 3d. If no precipitate is formed while the gas is evolved by ebullition, and the solution becomes strongly alkaline, effervescing with acids, alkaline bicarbonates existed in the original water.

Sulphuretted hydrogen is detected by the black precipitate produced by salts of lead or copper, and by the nauseous odour possessed by the water. If after boiling for a considerable period, a black precipitate is still produced by the metallic solutions, alkaline sulphurets are contained in the water.

Second Portion. For *sulphuric* and *hydrochloric acids*. —

Hydrochloric acid is shown by the white curdy precipitate produced by nitrate of silver, insoluble in nitric acid, but soluble in ammonia. To the solution filtered from chloride of silver add a solution of chloride of barium and a little nitric acid; a white precipitate indicates *sulphuric* acid.

Third Portion. *Iodine* and *bromine*. — Evaporate a large quantity of the water to a very small bulk, and divide into two portions. With one mix a little starch, and allow chlorine gas to fall on the surface of the liquid from a bottle of chlorine water. If a trace of *iodine* exists, it will be immediately recognised by the blue colour produced.

To discover *bromine*, add chlorine water to the other portion: if this element is present, a brown colour is developed, which is removed by agitation with ether. On standing for some time, the ether rises to the top, carrying with it all the bromine: add potash to the ethereal solution, evaporate to dryness, redissolve in water, and apply other tests for bromine.

Fourth Portion. For *nitric* and *boracic acids*. — Evaporate to dryness. If *nitric acid* is present, the dry salt deflagrates when heated strongly and touched with a glowing match.

When the dry residue is moistened with alcohol and a little sulphuric acid, if the alcohol burns with a green flame, *boracic acid* is present.

Fifth Portion. For *sulphurous acid*. — This acid is recognised by the action of iodic acid. (page 53.) Strong sulphuric acid causes an effervescence of sulphurous acid gas from a concentrated solution.

Sixth Portion. For *phosphoric acid*. — Sulphate of magnesia mixed with a little free ammonia causes a white crystalline precipitate of phosphate of magnesia and ammonia.

Seventh Portion. For *organic acids* (crenic, apocrenic, and puteanic). The dry residue of evaporation becomes carbonized when heated to redness, if it contains organic bodies.

To the preceding extended examination I subjoin a simpler and more cursory one, which will, in many cases, afford sufficient information on the nature of the water.

Different quantities of water are to be used for each test.

1. Oxalate of ammonia or binoxalate of potash produces a white precipitate of *oxalate of lime*.

2. Subphosphate of ammonia, a white precipitate of phosphate of *magnesia* and ammonia.

If oxalate of ammonia and subphosphate of ammonia give precipitates before but not after ebullition, the lime and magnesia exist entirely as carbonates. X

3. Yellow or red prussiate of potash gives a blue precipitate if *iron* is present. If the water is carbonated, and has been boiled, a few drops of hydrochloric acid should be added to redissolve the precipitate formed on ebullition.

4. Tincture of nut-galls, with a few drops of lime-water, gives a blackish brown colour, when a very minute quantity of *iron* exists. If this tincture and prussiate of potash give X

indications of iron after boiling for half an hour, when no acid has been added, the iron does not exist solely in the state of carbonate.

For *carbonic acid* apply the tests of litmus and of lime-water; for *sulphuretted hydrogen*, of acetate of lead; for *sulphuric acid*, of chloride of barium, and for *hydrochloric acid*, of nitrate of silver.

These directions for the qualitative analysis of mineral waters do not, however, indicate the actual salts which are present as they exist, but only their respective acids and bases. The arrangement of these in the water cannot be determined by any process of qualitative analysis, except by the application of a rule, which is not without exceptions, that the strongest acids are in combination with the strongest bases. But when the quantitative analysis of the water has been performed, the arrangement of the acids and bases can generally be ascertained with tolerable accuracy, from the difference which exists in the combining numbers of bodies. Thus, the quantities of sulphuric acid required for neutralization by the two bases, magnesia and potash, differ considerably; so that an amount of sulphuric acid having been found which is equivalent to the magnesia, and not to the potash, the former, and not the latter, is concluded to be in the state of sulphate. Another acid will probably be found whose amount will correspond to that of the potash, with which therefore the alkali was probably united.

SECTION VII.

ON THE USE OF THE BLOWPIPE IN QUALITATIVE ANALYSIS.

From its ready application and manageability, from its portability, convenience, and cheapness, and from the highly characteristic effects produced by it with chemical substances, the blowpipe is invaluable to the chemist as a subordinate or a confirmatory agent to the results previously

obtained or afterwards to be sought in humid analysis. By its

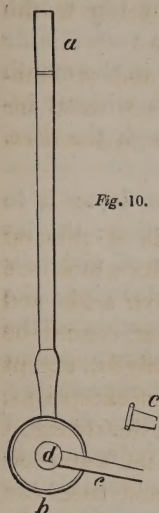


Fig. 10.

means we can raise the temperature of a small quantity of a substance almost instantaneously to a white heat; when this is done in different ways, as in a closed tube, in an open tube, alone or mixed with other bodies, or in different parts of the flame, we arrive at positive conclusions respecting the nature of the bodies operated on. This little instrument is simply a tube open at both extremities, the aperture in one being contracted so as to become capillary, through which a small stream of air is forced upon a flame. It would be out of place here to enter into details respecting the construction of the various kinds of blowpipes. A very convenient one is Voigt's, which is represented in the annexed figure. It consists of

a brass tube, with an ivory mouth-piece *a*, at one extremity, and at the other a circular box *b*, from which issues the small tube *c*, moveable round the centre *d*, by which any degree of obliquity may be given to the flame: *e* is a brass or platinum jet, which fits on the tube *c*. The only inconvenience attending this form is, that the tube *c* becomes loose by long usage. Griffin's (*fig. 11.*), which is of the same construction as Black's, is a cheaper and equally efficient instrument. It is made of japanned tin, with a brass nozzle.

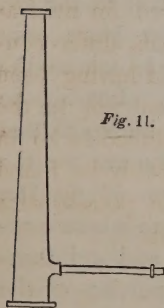


Fig. 11.

As the current of air which is supplied ought to be continuous, its production requires some attention and address. The air is not blown directly from the lungs, but is forced from the mouth by means of the cheeks. The difficulty consists in inspiring and expiring through the nose, while a continued stream escapes from the mouth. This may be attained by attention to the following directions: —Inflate the mouth fully, and then, with the lips firmly closed, and the back of

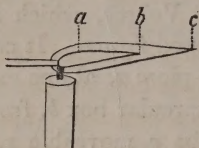
the mouth closed by the tongue, breathe freely through the nostrils. While the respiration proceeds, and the mouth is inflated, allow a little air to escape through a very minute opening between the lips, renewing the supply in the mouth by occasionally admitting air from the lungs without interfering with the process of respiration through the nose. A little practice renders the operation easy.

The effect of this current of air through a flame is to invert it, or rather to cause a double combustion: the interior, which before could not burn for want of air, now burns by means of the air forced through the blowpipe; the external atmosphere supplying air for the combustion of the exterior of the flame as before. This will be more apparent by reference to the annexed figures. *Fig. 12.* represents a

Fig. 12.



Fig. 13.



section of the ordinary flame of a candle: the interior cone (*a b*) is composed of combustible vapours or gases formed from the tallow, prevented from burning by having no access to the air, combustion taking place only on the exterior, *b*, where the

vapour is in contact with the atmosphere. Let air be admitted into the interior, as is done by the blowpipe, and these gases and vapours are burned, as they are on the exterior; the flame has then the appearance of *fig. 13.*, *a* representing the interior portion of the flame where combustion takes place. Between the points *b* and *c* inflammable gases still unburned exist; now this portion of the flame possesses properties very different to those possessed by the portions either beyond or within it. If oxide of copper be heated in this portion, oxygen is abstracted from the oxide by the combustible gases (which at this high temperature exhibit an intense affinity for oxygen), and metallic copper is produced: on the other hand, if metallic copper be heated beyond the point *c*, it is again oxidated, the oxygen being now supplied from the external atmosphere. From possessing such different properties, these portions of the

flame are called the oxidating and the deoxidating flames. The latter is also called the reducing flame. The chief oxidating effect is, however, produced beyond the flame, and this action is greater the farther the substance is from it, provided the temperature be maintained sufficiently high.

The apparatus to be used with the blowpipe can be but briefly described here. For a flame, where coal gas is available, that is to be preferred: the aperture of the burner is made of an oblong shape, instead of round, the current of air being blown lengthways. A candle having a large wick may also be employed; but it is inconvenient from the melting of one side by the bent flame and by the radiant heat of the ignited substance under examination. The oil lamp recommended by Berzelius consists of a tin cylinder about one inch in diameter, and four inches long, having a flat wick about three quarters of an inch broad. It is supported at one end by a retort-stand.

Various supports are used to hold the substance while exposed to the blowpipe flame, depending on the nature of the operation to which the matter is submitted. When heated alone, a pair of tongs or forceps, having small platinum points, a piece of platinum foil held by forceps, a platinum capsule, or a piece of dense charcoal, may be used. When heated with fluxes, as borax or microcosmic salt, a piece of small platinum wire is employed, bent twice at right angles at one extremity to retain the fused globule: a piece of charcoal or a bone-earth cupel is also employed for this purpose. Glass tubes, open or closed at one end, are used when it is suspected something volatile may be given off. None but hard German glass should be used for blowpipe experiments.

The principal operations to be performed are, —

1st. To heat the substance in a glass tube sealed at one end;

2d, in a tube open at both ends;

3d, alone; and,

4th, with fluxes or other reagents.

The chief objects of the *first* operation are, to ascertain whether a sublimate occurs, or whether the substance contains

any volatile matters; whether decrepitation takes place; whether the volatile products possess an acid or an alkaline reaction or are neutral; whether the substance becomes black on heating, showing the presence of organic matter, and the degree of fusibility of the substance. The tubes employed should be about two or three inches in length, and from one eighth to one fourth of an inch in diameter. The body under examination may occasionally require to be mixed with some reagent, as bisulphate of potash or charcoal.

The *second* operation is done with a view of ascertaining what effect a current of atmospheric air has on the substance at a high temperature: to know if a sublimate is produced; and if so, whether similar to that in the closed tube; and whether any peculiar odour is developed. By inclining the tube more or less, we have the means of regulating the current of air which passes: if heated when held nearly horizontal, very little air passes through, but the air increases in proportion as the tube is held more and more vertical.

In the *third* operation many objects are to be held in view. The degree of fusibility of the substance is observed; for which purpose a comparative scale has been established, consisting of the following minerals, in the order of their fusibility, beginning with the least fusible: sulphuret of antimony, natrolite, garnet, hornblende, felspar, and diallage. The changes exhibited by the substance must be noticed, as change of colour, intumescence, or deflagration. It must be ascertained whether the substance appears the same in the reducing as in the oxidating flame, and whether any volatile matters are evolved. An important object is to ascertain whether the substance produces any change on the colour of the blowpipe flame. The change, if any, is best seen when the matter just touches the flame at the top, about the middle of its length.

For this operation the substance is usually held in a pair of forceps having small platinum points.

The *fourth* operation is heating with reagents. Except in a few particular cases, the only reagents employed are borax,

microcosmic salt, and carbonate of soda. The object of heating with the two first, is to see what colour glass results when the substance is dissolved in the flux, both in the interior or reducing, and in the exterior or oxidating flame.

Fig. 14.



A platinum wire, bent as in the annexed figure, is employed in this operation for borax; but as microcosmic salt gives a very fluid bead when heated, a few additional bends must be made, when this salt is used, to prevent the melted drop from falling. Instead of the platinum wire, charcoal may be used to support microcosmic salt.

In fusing with carbonate of soda, the objects to be ascertained are, whether the substance gives a reduced metal, and whether it is soluble in the carbonate, giving a *glass*, or insoluble, giving a *slag*. A platinum wire is sometimes employed, but charcoal is the proper support for carbonate of soda; a dense piece should be selected, and a cavity made in it, from one-third to one-half of an inch in diameter, bored to about one-eighth of an inch in depth. The carbonate of soda, in fine powder, is kneaded into a lump with a little water, mixed with the substance for examination, and placed in the charcoal cavity. To ascertain whether a reduced metal has been formed by heating, the bead, when cold, is removed from the charcoal, and powdered with a little water, in an agate mortar. The particles of charcoal and soda are washed away by affusion, while any metallic particles present, subside to the bottom, and may be further examined as to colour, malleability, &c.

In the first three of the following tables are exhibited the most remarkable of the phenomena which metallic oxides present before the blowpipe; then the characters of some acids (in combination); after which tabular views are given of the successive operations (with their subdivisions) which are to be performed, and appearances to be looked for in the examination of an unknown substance. When, by following the processes of the latter tables, a clue to the nature of the substance is obtained, the result should be confirmed by examining whether it possesses all the characters which belong to that substance, as contained in the former tables.

I. *Behaviour of ALKALIES*

	Alone.	Colour of Flame produced by moist soluble Salts.	With Borax.
Potash.	Fusible.	Faint reddish or violet.	The red glass of borax and nick- el is changed to blue.
Soda.	Fusible.	Intense greenish yellow (cha- racteristic).	The red glass of borax and nickel is not changed to blue.
Lithia.	Fusible.	Crimson (cha- racteristic).	
Barytes.	Infusible. The hydrate and carbonate are fusible.	Pale apple green.	Colourless bead, opaque if satu- rated with ba- rytes.
Strontian.	Ditto.	Intense crimson (characteristic).	Ditto.
Lime.	Infusible, gives a strong light.	Reddish purple.	Ditto.
Magnesia.	No change.		Ditto.
Alumina.	Ditto.		Colourless glass, remains so when cold. Opaque with excess.
Glucina and Yttria.	Ditto.		Transparent glass; becomes milky by an intermittent flame.
Zirconia.	Infusible, gives a very powerful light.		Ditto.

and EARTHS.

With Microcosmic Salt.	With Carbonate of Soda on Charcoal.	Moistened with Solution of Nitrate of Cobalt.
A colourless bead, becomes opaque when cold, if saturated.	Is absorbed by the charcoal.	Brick or yellowish red while hot ; colourless when cold.
Ditto.	Not dissolved.	Black or grey.
Ditto.	Not dissolved ; is absorbed by the charcoal.	Ditto.
Ditto.	No action.	Flesh colour after strong heating (characteristic).
Transparent glass.	Undissolved.	A fine azure (characteristic).
As with borax.	No action.	Greyish black.
Scarcely transparent.	Ditto.	

II. *Behaviour of*

	Alone.		With Borax.	
	Oxidating Flame.	Reducing Flame.	Oxidating Flame.	Reducing Flame.
Antimony, oxide of.	Sublimes ; gives a greenish blue flame ; is reduced on charcoal.		Yellow glass while hot ; colourless when cold.	Grey from reduced metal.
Bismuth, oxide of.	Fusible ; dark brown while hot, yellow when cold on wire. Reduced on charcoal.		Colourless bead.	Ditto.
Cadmium, oxide of.	On platinum wire, no change. Is reduced on charcoal and volatilized, giving an orange red coating of oxide.		Yellowish glass, colourless when cold ; becomes opaque with an intermitting flame.	
Cerium, oxide of.	Protoxide becomes peroxide.		Red or orange when hot, pale when cold.	Colourless ; milk white if saturated.
Chromium, oxide of.	No change.		Red while hot, green when cold.	Green.
Cobalt, oxide of.	Ditto.		Deep blue (characteristic).	
Copper, oxide of.	Bromide and chloride give a blue flame ; most other salts give green.		Green while hot.	Brown red ; increased by the addition of tin.
	Fuses.	Reduced.		
Iron, peroxide of.	No change.	Blackens and becomes magnetic.	Red while hot ; pale when cold.	Bottle-green.
Lead, oxide of.	Gives a clear blue flame. Fuses to a fine orange glass ; is reduced on charcoal.		Yellow ; nearly colourless when cold.	
Manganese, oxide of.	Infusible. Protoxide becomes brown.		Amethyst, black if in excess ; assisted by nitre.	Colourless if quickly cooled.

METALLIC OXIDES. (a.)

With Microcosmic Salt.		With Carbonate of Soda.
Oxidating Flame.	Reducing Flame.	
Faint yellow glass : colourless when cold.	Grey from reduced metal.	Colourless glass on wire. Re- duced on charcoal. The reduced metal is very fusible and brittle, disengages a white smoke of oxide when heated in the air.
Brownish yellow while hot ; co- lourless when cold.	Reduced.	Reduced on charcoal. The reduced metal is brittle ; heated on charcoal it gives a yellow coating of oxide.
A transparent glass, opaque when cold, if saturated.		Does not melt on the wire. On charcoal it is reduced, and cadmium is volatilized. The charcoal acquires a red- dish brown coating (cha- racteristic).
As with borax.	Colourless.	Not dissolved.
Red while hot, green when cold, in both flames (characteristic).		In oxidating flame, dark orange when hot, opaque when cold. In reducing flame, opaque, becoming green on cooling.
Deep blue (characteristic).		Reduced on charcoal, giving a grey magnetic powder.
As with borax.		Green when hot ; colourless and opaque when cold. Is reduced on charcoal, giving a red malleable metal.
As with borax.	Green when hot, almost colourless when cold.	Reduced on charcoal, giving a magnetic powder.
Yellowish when saturated ; opaque when cold.		A transparent glass on wire, yellow and opaque when cold. Instantly reduced on charcoal.
As with borax (characteristic).		

III. *Behaviour of*

	Alone.	With Borax.	
		Oxidating Flame.	Reducing Flame.
Molybdic acid.	Fuses, giving a smoke in the open inclined tube and on platinum foil. Is reduced with difficulty on charcoal.	Colourless on platinum wire.	Brownish red on charcoal.
Nickel, oxide of.	No change.	Orange red ; almost colourless when cold.	Grey from reduced metal.
Silver, oxide of.	Instantly reduced.	Partly reduced and partly dissolved.	
		Milk white or opaline when cold.	Grey.
Tellurium, oxide of.	Gives the flame a green colour. Fuses and sublimes ; is easily reduced on charcoal.	Colourless.	Grey.
Tantalum, oxide of.	No change.	Colourless ; becomes opaque when cold by an intermittent flame.	
Tin, oxide of.	In oxidating flame, protoxide becomes peroxide. In reducing flame, is reduced by long heating.	Colourless.	
Titanium, oxide of.	No change.	Colourless ; becomes opaque on cooling.	First yellow, then amethyst, becoming darker on cooling.
Tungstic acid.	Blackens, does not fuse.	Colourless.	Orange, becoming darker as it cools. Tin produces an enamel.
Uranium, oxide of.	Ditto.	Yellow.	Green ; blackened by an intermittent flame.
Vanadium, oxide of.	On charcoal, fuses, and is partially reduced.	Yellow.	Brown when hot ; green when cold.
Zinc, oxide of.	Gives a strong whitish green flame. Slightly yellow while hot (by daylight) ; white when cold. Becomes green with solution of nitrate of cobalt.	Transparent glass, becoming milky by an intermittent flame.	Reduced ; the metal volatilizes.

METALLIC OXIDES. (b.)

With Microcosmic Salt.		With Carbonate of Soda.
Oxidating Flame.	Reducing Flame.	
Colourless on platinum wire.	Dark blue or black : green when cold.	Is reduced on charcoal.
Red ; colourless when cold.	As in oxidating flame. Tin destroys the colour.	Gives the reduced metal on charcoal. A magnetic powder.
Yellowish by daylight ; red by candle-light. Opalescent in ex.	Grey.	Reduced.
Colourless.	Grey.	On the wire a colourless glass, which becomes opaque on cooling.
Colourless glass.		Combination with effervescence ; no solution nor reduction.
Colourless.		Easily reduced on charcoal, giving a soft and very fusible metal.
Colourless.	Yellow while hot ; violet when cold.	Dissolves with effervescence, giving a yellowish glass, which becomes greyish white and opaque on cooling. Is not reduced on charcoal.
Very slightly yellow.	Very fine blue ; ox. of iron makes it blood-red ; this is prevented by tin.	Dark yellow glass ; becomes opaque on cooling. Is reduced on charcoal.
Yellowish while hot ; greenish when cold.	Green.	Insoluble.
As with borax in both flames.		Dissolves.
As with borax in both flames.		Insoluble. Is reduced on charcoal, which becomes covered with a sublimate of oxide.

BLOWPIPE TESTS TO DISTINGUISH ACIDS, OR THE
ELECTRO-NEGATIVE CONSTITUENTS.

Sulphates. — Mixed with the colourless and transparent glass of silica and soda, sulphates give, in the interior flame, a deep yellow or red colour, either immediately or on cooling. Heated with soda on charcoal, in the interior flame, the resulting mass blackens metallic silver when moistened.

Nitrates. — Those which are fusible deflagrate on charcoal. Infusible nitrates, heated to redness in a glass tube give off nitrous vapours. All nitrates give deep red vapours when mixed with bisulphate of potash and heated.

Borates. — Fused with carbonate of potash on charcoal, and moistened with sulphuric acid and alcohol, a green flame is produced. Mixed with a flux of one part of fluor spar and four and a half of bisulphate of potash, borates give a dark green flame for an instant, when held at the point of the blue flame on a platinum wire.

Phosphates. — To detect phosphoric acid, fuse the substance with boracic acid on charcoal; when fused, introduce the extremity of a steel wire and heat strongly in the interior flame. Borate and phosphuret of iron are formed; on breaking the fused mass (wrapped in paper) in a mortar, the phosphuret of iron is perceived, having the appearance of a metallic button, which affects the magnet and is brittle.

Moistened with sulphuric acid, and held in the platinum tongs at the point of the blue flame, phosphates give a pale green colour to the outer flame.

Silicic acid is characterized by dissolving easily in carbonate of soda with effervescence, giving a transparent and colourless bead. It is slowly dissolved in borax, giving a clear glass.

Seleniates and *selenites* give the characteristic odour of selenium, with soda on charcoal, in the interior flame. They behave like sulphates with the glass of silica and soda.

Arseniates and *arsenites.* — On charcoal, in the interior

flame, they give the characteristic alliaceous odour of arsenic. Mixed with black flux and heated in a glass tube closed at one end, a brilliant sublimate of metallic arsenic is formed, crystalline in its interior surface. It may be chased up the tube, becoming slowly oxidized. If the bottom of the tube be cut off, and the sublimed metal heated, it is immediately converted into arsenious acid, which condenses as a white crystalline sublimate.

Sulphurets. — Heated in an open inclined tube, or on charcoal, they evolve the odour of sulphurous acid, which acid bleaches moist Brazil-wood paper. Sulphurets behave as sulphates with the glass of silica and soda. After fusion with soda on charcoal, the moistened globule blackens metallic silver.

Seleniurets, in the exterior flame, evolve a strong odour resembling putrid horse-radish. Heated in the open tube, metallic selenium sometimes condenses. With the glass of silica and soda, seleniurets behave as seleniates and sulphates.

Antimoniurets give a sublimate of oxide of antimony when roasted in the open tube.

Tellurets give, when roasted in the inclined open tube, a sublimate of a white powder, which, when heated, fuses into colourless drops before it sublimes.

Chlorides, added to a glass of microcosmic salt, saturated with oxide of copper (or held by a very small brass instead of a platinum wire), and suddenly heated, communicate a bright colour to the flame.

Iodides, with the glass of microcosmic salt and oxide of copper, give a fine emerald green flame. Heated with bisulphate of potash in a glass tube closed at one end, iodine vapour is evolved.

Bromides, with the glass of microcosmic salt and oxide of copper, give a greenish blue flame. Heated with bisulphate of potash, bromine vapour is expelled.

Fluorides. — Those which contain water, give off hydrofluoric acid when heated in a tube closed at one end. All fluorides, when mixed with bisulphate of potash, or with

microcosmic salt previously fused, and heated at the lower extremity of an open glass tube, evolve hydrofluoric acid, known by its corrosive action on glass, and its property of bleaching Brazil-wood test paper.

OPERATIONS TO BE PERFORMED IN THE EXAMINATION OF AN UNKNOWN SUBSTANCE.

FIRST OPERATION. — Heating in a glass tube closed at one end.	(a) Volatile salts.	Ammoniacal salts.	{ They sublime without decomposition, unless they contain a fixed acid.
		Most salts of mercury.	{ Heated with dry carbonate of soda, give metallic mercury.
		Many chlorides of fixed oxides.	{
	(b) Oxides and acids sublimed.	Oxide of antimony.	{ Sublimes in shining needles.
		Arsenious acid.	{ White octahedral crystals.
		Arsenic acid.	{ Becomes converted into oxygen gas and arsenious acid.
		Oxide of tellurium.	{ Sublimate not crystalline.
		Osmic acid.	{ Recognised by its odour.
	(c) Volatile acids and ammonia.	{ Ascertained by their action on red and blue litmus paper.	
		{	
	(d) Volatile metals.	Tellurium.	{ Volatile only at a bright red heat.
		Cadmium.	{ Gives a yellowish-red coating to charcoal when heated on it in the open air.
		Mercury.	{ Condenses in liquid drops.
		Arsenic.	{ Interior surface of the sublimate is crystalline. Odour of garlic evolved.
	(e) Water.	{ Condenses on the sides of the tube.	
	(f) Sulphur.	{ A yellow sublimate.	
	(g) Selenium.	{ Characterized by its odour.	
	(h) Organic bodies.	{ Become black, leaving a residue of charcoal.	

SECOND OPERATION. — Heating alone, in the open air.

(a)	Fusible oxides and acids.	{	Antimony, oxide of; bismuth, ox. of; copper, ox. of; lead, ox. of; molybdic acid; tellurous acid; vanadic acid.
(b)	Infusible oxides and acids.	{	Alumina; antimonious acid; barytes; cadmium, ox. of; cerium, perox. and protox. of; chromium, ox. of; cobalt, ox. of; glucina; iron, perox. of; lime; magnesia; manganese, perox. of; nickel, ox. of; silica; strontian; tantallic acid; tin, perox. and protox. of; titanlic acid; tungstic acid; uranium, perox. and protox. of; yttria; zinc, ox. of; zirconia.
(c)	Colour flame.	{	of Blue. Green. Red. Yellow. { Chloride and bromide of copper; lead; arsenic; selenium; antimony. Zinc; barytes; boracic acid; tellurium; copper; phosphoric acid; ammonia. Lime; strontian; lithia; potash. Soda; water.
(d)	Volatile bodies.	{	Metals. Produce a sublimate on charcoal. Give no sublimate on charcoal — mercury; osmium. { Antimony; arsenic; bismuth; cadmium; lead; tellurium; tin; zinc. Compounds of mercury. Oxides of volatile metals in the reducing flame. Sulphur; selenium; iodine; bromine. Many chlorides and iodides.
(e)	Change of colour on ignition.	{	Red oxide of mercury, minium and most chromates are black when hot, and red or yellow when cold. Oxide of zinc and titanlic acid are yellow when hot, and white when cold.

[Observe the dissimilar action of the exterior and interior flames.]

THIRD OPERATION.—Heating in a glass tube open at both ends.

(a) White sublimates.

Oxide of antimony.

{ Formed during the roasting of metallic antimony, sulphuret of antimony, and of metallic antimonurets; also by heating oxide of antimony.

Arsenious acid.

{ Formed by the roasting of arseniurets.

Oxide of bismuth.

{ Formed by the roasting of sulphuret of bismuth and alloys of bismuth. The sublimate melts into brownish or yellowish drops when heated.

Chloride of lead.

Melts when heated.

Oxide of lead.

{ Accompanying bodies which form volatile oxides or acids.

Selenite of lead.

From roasting the seleniuret.

Sulphate of lead.

From roasting the sulphuret.

Mercury.

From most mercurial compounds.

Molybdic acid.

{ Sublimes partly in the state of shining pale yellow crystals.

Oxide of tellurium.

(b) Odours.

Sulphurous acid.

From metallic sulphurets.

Selenium.

{ From metallic seleniurets. A red sublimate of selenium is sometimes formed.

Arsenic.

From a few arseniurets only.

FOURTH OPERATION. — Heating with reagents.

1. Heating with carbonate of soda.
- (a) Fusible with soda on platinum wire in the oxidating flame. { Oxide of antimony ; oxide of chromium ; oxide of cobalt ; oxide of copper ; oxide of lead ; oxides of manganese ; molybdic acid ; silica ; oxide of tellurium ; titanitic acid ; tungstic acid ; vanadic acid.
 - (b) Fusible with soda on charcoal, giving a clear bead. { Silicic acid, with evolution of carbonic acid gas.
Titanic acid. { Giving a yellow glass white hot, opaque when cold.
 - (c) Metals whose compounds are reducible with soda on charcoal in the reducing flame. { The oxides form a sublimate on the charcoal. { Antimony ; arsenic ; bismuth ; cadmium ; lead ; selenium ; tellurium ; zinc.
No sublimate is deposited on the charcoal. { Cobalt ; copper ; gold ; iron ; molybdenum ; nickel ; platinum ; silver ; tin ; tungsten.
 - (d) Neither fused nor reduced with soda on charcoal. { Alumina ; barytes ; oxides of cerium ; glucina ; lime ; magnesia ; strontian ; tantalitic acid ; thorina ; oxides of uranium ; yttria ; zirconia.
 - (e) Examine for sulphur and selenium by the glass of silica and soda.

2. Colours of beads with borax (while hot).

In oxidating flame.		In reducing flame.	
Colourless	Alumina	Colourless	Alumina
	Antimony		Barytes
	Barytes		Cadmium
	Bismuth		Cerium
	Cadmium		Glucina
	Glucina		Lime
	Lime		Magnesia
	Magnesia		Manganese
	Molybdic acid		Silica
	Silicic acid		Strontian
	Strontian		Tantalitic acid
	Silver		Thorina
	Tantalitic acid		Tin
	Tellurium		Yttria
	Thorina		Zinc
	Tin		Zirconia.
	Titanic acid	Reddish brown	Copper
	Tungstic acid		Molybdic acid.
	Yttria	Yellow	Tungstic acid
	Zinc		Titanic acid.
	Zirconia.		

In oxidating flame.		In reducing flame.	
Red	{ Cerium { Chromium { Iron { Nickel.	Green	{ Chromium { Iron { Uranium { Vanadium.
Yellow	{ Lead { Uranium { Vanadium.	Blue	{ Cobalt.
Green	{ Chromium { (when cold) { Copper.	Grey	{ Antimony { Bismuth { Nickel. { Silver { Tellurium.
Violet	Manganese.		
Blue	Cobalt.		

3. Colours of beads with microcosmic salt (while hot).

In oxidating flame.		In reducing flame.	
Colourless	{ Alumina { Antimonious acid { Barytes { Cadmium { Glucina { Lead { Lime { Magnesia { Molybdic acid { Strontian { Tantallic acid { Tellurium { Thorina { Tin { Titanic acid { Tungstic acid { Yttria { Zinc { Zirconia.	Colourless	{ Alumina { Barytes { Cadmium { Cerium { Glucina { Lime { Magnesia { Manganese { Strontian { Tantallic acid { Thornia { Tin { Yttria { Zinc { Zirconia.
Red	{ Cerium { Iron { Nickel { Chromium.	Red	{ Antimonious acid with iron. { Nickel { Titanic acid with iron { Tungstic acid with iron.
Yellow	{ Bismuth { Silver { Vanadium.	Yellow	{ Titanic acid (violet when cold).
Green	{ Copper { Molybdic acid { Uranium { (Chromium when cold).	Green	{ Chromium { Iron { Molybdic acid { Uranium { Vanadium (when cold).

	In oxidating flame.		In reducing flame.
Violet	Manganese.		
Blue	Cobalt.	Blue	{ Cobalt Molybdic acid Tungstic acid.
		Brown	Copper.
		Grey	{ Antimony Bismuth Lead Silver Tellurium.

SECTION VIII.

ANALYSIS OF GASES.

I. PROPERTIES OF GASES.

Oxygen -	} Re-inflame a glow-				
Nitrous oxide		ing taper.	-		
Ammonia -		-	-		
Hydrochloric acid -		-	-		
Chlorine -		-	-	} Soluble in	
Sulphurous acid -		-	-		
Carbonic acid -		-	-		} Absorbed by potash.
Sulphuretted hydrogen		-	-		
Cyanogen -		-	-		
Olefiant gas -		-	-		
Phosphuretted hydrogen		-	-		} Combustible.
Arsenietted hydrogen		-	-		
Carbonic oxide -		-	-		
Hydrogen -		-	-		
Light carburetted hydrogen		-	-		

II. QUALITATIVE ANALYSIS OF A MIXED GAS; ITS CONSTITUENTS BEING CONTAINED IN THE FOLLOWING LIST.

- | | |
|---------------------------|--------------------------------|
| 1. Carbonic acid | 10. Oxygen |
| 2. Hydrochloric acid | 11. Nitrous oxide |
| 3. Chlorine | 12. Nitric oxide |
| 4. Sulphurous acid | 13. Light carburetted hydrogen |
| 5. Sulphuretted hydrogen | 14. Olefiant gas |
| 6. Cyanogen | 15. Carbonic oxide |
| 7. Phosphuretted hydrogen | 16. Hydrogen |
| 8. Arsenietted hydrogen | 17. Nitrogen. |
| 9. Ammonia | |

(A.) Into the dry gas contained over mercury, introduce moistened sticks of potash; withdraw the potash, and dissolve it in water.

The first six of the list are absorbed.

- | | | |
|--|---|---|
| 1. <i>Carbonic acid.</i> | { | Remove the more soluble gases from a portion of the original gas by water, and apply the test of lime-water to the residue. |
| 2. <i>Hydrochloric acid,</i>
and
3. <i>Chlorine.</i> | | Neutralize the solution of the potash by pure nitric acid, and test with nitrate of silver. Chlorine and hydrochloric acid are distinguished from each other by the power of the first to bleach vegetable colours. |
| 4. <i>Sulphurous acid.</i> | { | Peroxide of lead agitated in the original gas absorbs sulphurous acid. |
| 5. <i>Sulphuretted hydrogen.</i> | | The solution of the potash produces a black precipitate in salts of lead. |
| 6. <i>Cyanogen.</i> | { | To the solution of the potash apply the test of peroxide and protoxide of iron (Scheele's test), page 49. |

(B.) After the absorption of the potash, remove the remaining gas to another jar, and introduce a solution of nitrate of silver. Nos. 7, 8, and 9. are absorbed.

- | | | |
|-----------------------------------|---|--|
| 1. <i>Phosphuretted hydrogen.</i> | { | Combustible ; producing white fumes of phosphoric acid ; is sometimes spontaneously inflammable. |
| 2. <i>Arsenietted hydrogen.</i> | | Gives a volatile deposit of metallic arsenic when passed through a red-hot tube. |
| 3. <i>Ammonia.</i> | { | Known by its great solubility in water, and its alkaline reaction. |

(C.) The gases which remain after the action of potash, and of nitrate of silver, are, Nos. 10, 11, 12, 13, 14, 15, 16, 17.

1. Introduce a stick of phosphorus, and allow it to remain so long as any contraction occurs. *Oxygen* is absorbed.*

2. If the gas supports combustion before, but not after, agitation with water, it contains *nitrous oxide*.

(D.) Into a *portion* of the remaining gas contained in a tube through which the electric spark can be passed †, pass up an equal volume of pure *oxygen*.

1. The production of ruddy fumes shows the presence of *nitric oxide*.

2. Pass the electric spark ; if explosion takes place, Nos. 13, 14, 15, and 16. may be present.

* Phosphorus does not act upon oxygen in the presence of olefiant gas, unless the pressure be greatly reduced. Phosphorus is likewise without action on perfectly pure oxygen.

† Such as Dr. Ure's eudiometer, of which a figure is given under the subject of Eudiometry.

(E.) To the other portion of the gas (not mixed with oxygen), add its own bulk of *chlorine*, and keep in a dark place.

Contraction and formation of oily drops, show the presence of *olefiant gas*.

Withdraw the excess of chlorine by agitation with potash, and heat *potassium* in the residue so long as contraction occurs.*

Carbonic oxide is absorbed.

To the residue add an equal bulk of oxygen, and ignite by the electric spark. If explosion takes place, both light carburetted hydrogen and hydrogen may have been present. *Carburetted hydrogen* produces its own volume of carbonic acid. By ascertaining, (1.) the volume of the gas before adding oxygen ; (2.) the amount of oxygen added ; (3.) of carbonic acid produced ; and (4.) of oxygen in excess (by removing it afterwards by phosphorus), the presence and quantity of *hydrogen* may be calculated. (See following table.)

As nitrous oxide interferes with the above process, it must be previously removed by agitation with water. If any gas remains after detonation and absorption of carbonic acid by potash, and of oxygen by phosphorus, this must be *nitrogen*.

* This operation is best performed in a glass tube closed at one end, about eight inches in length, with one inch and a half from its top recurved to hold the potassium.

III. ANALYSIS OF COAL GAS (QUALITATIVE AND QUANTITATIVE).

Mix the gas with half its bulk of chlorine in the dark.

<i>Olefiant gas and vapours of hydrocarbons</i> are absorbed.	Remove the excess of chlorine by potash, and heat potassium in the residue contained in a recurved tube over mercury.
<i>Carbonic oxide</i> is absorbed.	Into the remainder introduce a known measure of oxygen (not less than twice the volume of the gas), and detonate. Note the quantity of gas, and introduce potash.
Carbonic acid equal in bulk to the quantity of <i>light carburetted hydrogen</i> is absorbed.	The residue consists of nitrogen and the excess of oxygen. Introduce phosphorus to absorb oxygen, that the whole <i>amount of oxygen employed in the combustion</i> may be found. From this deduct twice the volume obtained of light carburetted hydrogen; twice the bulk of the remaining oxygen represents that of the <i>hydrogen</i> . The residuary gas must be <i>nitrogen</i> .

SECTION IX.

DETECTION OF POISONS IN ORGANIC MIXTURES.

When an inorganic poison is presented for examination unmixed with organic matters, its composition may be ascertained by processes already described; but if the poison is to be looked for in the contents of the stomach, or any such organic mixture, its properties may have become so masked, its composition so altered, and the usual action of reagents so restrained, as to require a modification of the processes employed where organic matters are absent. In general, it is not convenient, in these examinations, to follow the course recommended in mineral qualitative analysis, of determining the presence or absence of certain classes of bodies, and afterwards proceeding to individual substances, but better to seek at once for a particular substance, by the application of its appropriate test. This plan is adopted for two reasons: first, because the act of poisoning is almost always committed with a single substance; and second, because there usually exist evidences, of a physiological or circumstantial character, which have excited a suspicion of the true nature of the poison; the appropriate tests for which are then applied.

The substances for the discovery of which, in organic mixtures, processes will be described, are the following:—

- | | |
|-------------|-----------------------|
| 1. Arsenic | 6. Nitric Acid |
| 2. Mercury | 7. Sulphuric acid |
| 3. Antimony | 8. Hydrochloric acid |
| 4. Copper | 9. Oxalic acid |
| 5. Lead | 10. Hydrocyanic acid. |

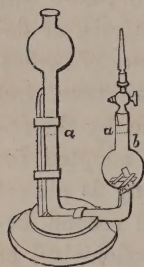
1. *Arsenic.*

The ordinary liquid tests for arsenic can seldom be sufficiently depended on in organic solutions. The only unequivocal test of the presence of arsenic is, then, the pro-

duction of the metal itself, by processes of reduction. This reduction is performed in two different ways: first, by the formation and subsequent decomposition of *arsenietted hydrogen gas*; and second, by the formation and reduction of the *sulphuret of arsenic* by means of *black flux*, a mixture of charcoal and carbonate of soda or potash.

The principle of the first method (Marsh's test), which is the simplest and easiest, is, that whenever hydrogen gas is evolved in contact with an arsenical compound, soluble or insoluble, the arsenic unites with hydrogen, forming arsenietted hydrogen gas, in which the existence of arsenic is easily demonstrated. The arrangement represented in

Fig. 15



the figure was contrived by Mr. Marsh, for the operation adverted to. The stopcock, which can be fitted to the tube by means of a cork, being removed, a fragment of pure zinc is introduced into the lower bulb, and dilute sulphuric acid poured upon it, up to the mark *a*. Hydrogen gas is evolved, with which the whole limb *b* is soon filled on closing the stopcock, the acid liquid being then forced into the upper bulb. As the zinc of commerce

almost always contains some arsenic, it is necessary to examine whether the hydrogen is free from it, or not. For this purpose the gas is kindled at the jet, and allowed to burn with a small flame. On holding the bottom of a porcelain bason or a stoneware plate *in the flame*, a black spot, of a bright metallic lustre, will immediately be formed on it, in the centre of the flame, if the gas contains any, even a very small amount of arsenic. Another method of discovering the presence of arsenic in the gas, is to hold a piece of coloured glass *over the flame*, when the glass becomes covered with a white sublimate of arsenious acid, if any arsenic is present. If the hydrogen gas presents these appearances, some arsenic must have existed either in the acid or the zinc; but if it does not, pour off some of the dilute sulphuric acid, and mix with the remainder a portion of the liquid to be tested. Proceed as before to collect the gas, and burn it

against a stoneware plate, and under a piece of coloured glass, as before directed.

Instead of burning the gas in the way described, it may be passed through a hard glass tube, a portion of which is heated red hot by a spirit-lamp, by which all the arsenic is reduced and condensed in the tube, near the flame of the

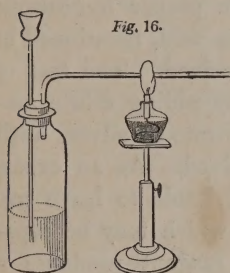


Fig. 16.

lamp. For this purpose, a bottle similar to that represented in the annexed figure may be employed. The funnel is for adding more acid or suspected liquid. This modification of the previous process is more manageable, and equally satisfactory. It may be left in action without requiring the constant presence of the operator; and the whole amount of

arsenic originally contained in the liquid operated on, may be obtained without loss. It affords, moreover, the means of distinguishing between arsenic and antimony, these metals being easily confounded by the test of burning the gas at the jet. Antimony forms with hydrogen a gaseous compound corresponding to arseniетted hydrogen, which also gives, on burning, a white sublimate, and a metallic deposit in the interior of the flame on a stoneware plate. But when antimonietted hydrogen is passed through a red-hot tube, the reduced antimony is deposited much nearer the part where heat is applied than arsenic is, and, unlike the latter metal, on both sides of the heated part. By these characters, antimonietted hydrogen and arseniетted hydrogen may be distinguished.

When the ordinary apparatus with the jet is employed, a good method of discriminating between antimony and arsenic is by collecting and examining the white sublimate formed by the combustion of the gas. For this purpose, the sublimate is most conveniently collected by condensing it within a glass tube open at both ends, held slightly oblique. This tube may be about six inches in length, and half an inch in diameter. When the sublimate produced by the combustion

of twice the contents of a bulb has been obtained, it may be treated with a little water, to which a single drop of solution of ammonia has been added, when it dissolves, if it is arsenious acid, giving a solution in which arsenic is easily recognised by the two principal fluid tests for arsenious acid, — namely, ammonio-nitrate of silver and ammonio-sulphate of copper. If the metal is antimony, the proper indications which these reagents give with arsenious acid, mentioned in the tables, pp. 42. and 45., are not perceived. The test of ammonio-nitrate of silver is prepared by adding dilute ammonia very gradually to a solution of nitrate of silver, until the oxide at first precipitated is redissolved. As an excess of ammonia would be injurious, it is advisable to leave undissolved a trace of the oxide of silver, which may be separated afterwards by filtration. This reagent produces a precipitate of the yellow arsenite of silver, as well in free arsenious acid as in a solution of an arsenite in water. The test of ammonio-sulphate of copper is prepared in a similar manner, with ammonia and a solution of sulphate of copper.

A sublimate of metallic arsenic may also be distinguished, and even separated from metallic antimony, by a solution of chloride of soda, or of chloride of lime, which dissolves the arsenic, but leaves the antimony untouched. (Bischoff.)

In the reduction of the sulphuret of arsenic to the metallic state, the other process of reduction, the following operations are necessary : —

“ I. Preparation of the fluid : —

1. Boil the matters with water and a few drops of nitric acid.
2. Strain through calico.
3. Precipitate animal matter by an excess of nitrate of silver, and subsequent addition of common salt.
4. Filter through paper.

“ II. Precipitation of the sulphuret of arsenic : —

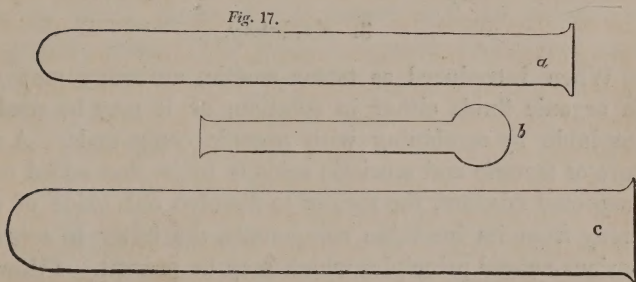
1. Transmit a stream of sulphuretted hydrogen through the liquid for half an hour.
2. Heat the liquid in an open vessel for a few minutes, to cause the precipitate to separate.

3. Wash the precipitate by affusion of water acidulated with hydrochloric acid, and subsidence.
4. Dry the precipitate at a temperature not exceeding 300° .

“III. Reduction of the sulphuret of arsenic: —

1. Mix the dried precipitate intimately with twice its weight of dry black flux (carbonate of potash and charcoal), and heat to redness in a glass tube, of the form and size of *a* or *b* exhibited below.
2. Heat slowly a particle of the metallic crust in a glass tube, *c*, and observe the formation of a white crystalline sublimate of arsenious acid.”

(Graham's *Elements of Chemistry*, pp. 633, 634.)



A solution of *arsenic acid* is precipitated by sulphuretted hydrogen with less facility than a solution of arsenious acid; a trace of arsenic acid may even effectually resist decomposition by sulphuretted hydrogen. When the arsenic, therefore, can be contained in a suspected liquid in the state of arsenic acid, it should be reduced to the state of arsenious acid before transmitting sulphuretted hydrogen. This may be readily effected by sulphurous acid at a boiling heat. (Wöhler.) An aqueous solution of sulphurous acid is mixed with the suspected liquid, and the mixture boiled; by this, the arsenic acid is reduced, while the excess of sulphurous acid is expelled in the ebullition. Sulphuretted hydrogen may then be passed through the solution, and the precipitated sulphuret treated in the usual manner.

2. *Mercury.*

The mixture suspected to contain mercury is to be evaporated almost to dryness, and the organic matter destroyed by boiling the residue with aqua regia so long as red fumes are evolved. Evaporate nearly to dryness, to expel most of the excess of acid, dilute with water, and filter. The solution thus obtained may be treated either with sulphuretted hydrogen, and the precipitated sulphuret subsequently reduced by heating in a small glass tube with carbonate of soda, or the solution may be mixed with protochloride of tin, and the precipitated metallic mercury collected and sublimed in a glass tube. (p. 37.)

3. *Antimony.*

When introduced as tartar emetic, antimony may exist in organic fluids either in solution, or it may be rendered insoluble by combining with organic compounds. A mixture of tartaric and muriatic acids is to be first added to the suspected mixture, the former to dissolve out oxide of antimony from its insoluble compounds, the latter to coagulate various animal principles which may be present. Filter and transmit sulphuretted hydrogen gas through the solution; an *orange-coloured* precipitate of sulphuret of antimony will be formed, if that metal be present. Collect the precipitate, dry and heat it in a small capsule, with strong hydrochloric acid; it is then dissolved, with evolution of sulphuretted hydrogen: evaporate off most of the excess of hydrochloric acid, and pour the liquid residue into pure water, on which a white precipitate of oxichloride of antimony will be produced. (p. 33.)

4. *Copper.*

This metal, like the preceding, may be present in an organic mixture in two states; in solution, and in a state of combination with organic principles, forming insoluble compounds. As acetic acid dissolves out the oxide of copper

from most of these organic compounds, the mixture, after being boiled, should be treated with that acid, diluted and filtered. Sulphuretted hydrogen gas is now passed through the solution, when, if copper is present, a black or brown precipitate of the sulphuret is formed, which, after boiling the liquid, is to be washed by affusion and subsidence. The precipitate is then collected in a porcelain capsule, heated to redness with access of air, to destroy any organic matter with which it may be mixed, and treated with nitric acid to dissolve the copper. The solution then obtained may be tested with ammonia, or any other characteristic test of copper. (p. 33.) If necessary to examine the portions of the suspected mixture insoluble in acetic acid, the matter should be well dried, and heated to redness in a porcelain crucible, by which most of the copper will be reduced to the metallic state. The residue, treated with nitric acid, affords a solution to which the usual tests of copper may be applied.

5. *Lead.*

Add a little nitric acid to the suspected mixture before filtration, to dissolve the compounds of oxide of lead and organic matters, and transmit sulphuretted hydrogen gas through the filtered liquid. Boil and collect the precipitate on a filter. To prove the existence of lead in the precipitate, digest it in moderately strong nitric acid, with the assistance of a very gentle heat, by which the lead will be dissolved, while almost all the sulphur remains unacted on. If strong nitric acid is employed, the sulphur will be converted into sulphuric acid, and the insoluble sulphate of lead formed. The acid solution of nitrate of lead must be poured off from the sulphur, evaporated to dryness, and redissolved in water. This solution can then be examined by chromate of potash, or any other test of lead. (p. 37.)

Instead of digesting the sulphuret of lead in nitric acid, it may be examined before the blowpipe, by heating with soda on charcoal, when it gives a globule of metal easily recognised

as lead, by its softness, ready fusibility, and the peculiar lustre of a freshly cut surface.

6. *Nitric acid.*

Neutralize the liquid for examination with potash, filter, and evaporate to obtain crystals of nitre. To the salt thus obtained, the test of protosulphate of iron and sulphuric acid may be applied (page 51.); or the crystals may be mixed with sulphuric acid and copper filings, when ruddy fumes of peroxide of nitrogen are perceived arising from the nitric oxide evolved. If much organic matter is present, the nitrate of potash will not crystallize; in which case the dry residue of evaporation is redissolved, and a solution of acetate of silver added, which not only removes organic matters, but also decomposes the chloride of sodium present in almost all organic liquids, and which would interfere when the crystallized salt is treated with sulphuric acid, by producing free chlorine. After precipitation by acetate of silver, filter, and evaporate to obtain crystals. (Christison.)

7. *Sulphuric acid.*

Although introduced in the state of oil of vitriol, sulphuric acid may soon exist in the form of a neutral sulphate, from coming in contact with bases or carbonates, or from the gradual formation of ammonia through decomposition of the animal matters. This acid, therefore, when suspected, must be looked for as well in the free as in the combined state. The existence of a free acid in considerable quantity having been ascertained by blue litmus paper, if the liquid does not give the usual appearance presented by sulphuric acid with a salt of barytes (page 52.), in consequence of the presence of organic matters, it must be distilled to dryness in a proper retort and receiver, the heat being increased to dull redness towards the close. The receiver should contain a dilute solution of ammonia, to arrest all the sulphurous acid which is formed by the action of the organic substances on sulphuric acid. On treating the distilled product with

aqua regia, the sulphurous acid is again converted into sulphuric acid, the presence of which is then easily ascertained by chloride of barium.

If the sulphuric acid is in a state of combination with bases, the liquid suspected to contain it may be filtered, and the ordinary tests applied. In examining organic mixtures for sulphuric acid, it is always necessary to discriminate between the small quantity naturally contained in these mixtures, and the large excess which should be found when sulphuric acid is really administered with a criminal intention. To do this satisfactorily, a quantitative analysis must generally be had recourse to.

8. *Hydrochloric acid.*

Nitrate of silver, the ordinary test for hydrochloric acid, cannot be applied to the contents of the stomach, from the constant presence of chlorides, as well as of organic matters which precipitate salts of silver. The mixture suspected to contain hydrochloric acid must be cautiously distilled to dryness in a chloride of calcium bath, composed of about equal parts of that salt and water. The test of nitrate of silver with nitric acid (page 49.) is then applied to the product of the distillation. If the subject of examination is the contents of the stomach, and a mere trace only is detected, no conclusion as to poisoning can be drawn, as this may have actually existed as an ingredient of the gastric juice.

9. *Oxalic acid.*

The mixture suspected to contain this acid is first filtered and mixed with carbonate of potash. The oxalic acid is then precipitated by a solution of acetate of lead; the precipitate (partly oxalate of lead) is collected on a filter, washed, diffused in water, and decomposed by sulphuretted hydrogen: sulphuret of lead and free oxalic acid are formed. The mixture is then filtered, heated to expel sulphuretted hydrogen, and a salt of lime and nitrate of silver are applied as tests (page 50.)

If lime or magnesia has been administered as an antidote, this process must be somewhat modified. The suspected mixture is allowed to stand some time to permit the insoluble oxalate to subside, and the supernatant liquid decanted. The insoluble residue is now boiled with a solution of carbonate of potash, and filtered. By this operation there are formed a carbonate of the earth and the oxalate of potash. Add to the solution a slight excess of nitric acid, filter, render the liquid alkaline by carbonate of potash, and filter again. The object of these operations is to get rid of animal matter, some portion being removed at each step. The alkaline solution of oxalate of potash is now precipitated by acetate of lead, and the precipitated oxalate treated as above by sulphuretted hydrogen.

10. *Hydrocyanic acid.*

If the organic mixture is of such a nature as to prevent the detection of this acid by its usual tests, recourse must be had to distillation. The mixture is first filtered, mixed with sulphuric acid, to combine with all the ammonia which may have been produced by the decomposition of organic matters, and then distilled in a water-bath until one eighth has been collected. To this product the tests of nitrate of silver and of potash and protosulphate of iron (page 49.) are applied.

SECTION X.

QUALITATIVE ANALYSIS OF URINE, AND DISCRIMINATION OF URINARY CALCULI.

ANALYSIS OF URINE.

(*Abridged from the "Traité de Chimie" of Berzelius.*)

The substances to be looked for are — uric acid, albumen, colouring matter of the bile, urea, phosphate of lime, phosphates of the alkalies, lime, sulphuric acid, phosphoric acid, fibrin, caseous matter, hydrochloric acid, mucus, free acid and alkali, and sugar.

The following are the reagents to be used, with their applications: —

1. *Nitric acid*. This is employed to detect uric acid, urea, albumen, and the colouring matter of the bile. It is known whether *uric acid* is present by adding a few drops of nitric acid to half a pint of urine, and allowing it to stand for twelve hours, when uric acid will be deposited on the sides of the vessel.

To detect *urea*, evaporate the urine to one eighth of its original bulk, and add half its reduced volume of nitric acid, when immediately, or on standing a short time, crystals of nitrate of urea are deposited. Nitric acid gives a floccy white precipitate with *albumen*, which is soluble in caustic potash after being washed, and is not reprecipitated by acetic acid.

2. *Ammonia* precipitates the *phosphate of lime* held in solution by the free acid of the urine.

3. *Lime-water* shows the presence of *alkaline phosphates*, by the precipitate of phosphate of lime which it produces.

4. *Oxalate of ammonia* is employed to precipitate the *lime* contained in urine. If ammonia is afterwards added, the *ammoniacal phosphate of magnesia* may be precipitated. Should no precipitation take place, add a solution of phosphate of soda, to ascertain whether this is owing to the absence of magnesia or that of phosphoric acid.

5. *Acetate of barytes* is used to indicate *sulphuric acid*. The urine must be slightly acidified by acetic acid.

6. *Neutral acetate of lead* may precipitate the *chloride* and the *phosphate* of lead: distinguish these by the blowpipe.

7. *Solution of alum* causes a troubling in urine that contains *albumen* or *fibrin* in solution.

8. *Chloride of mercury* (corrosive sublimate) gives no precipitate in acidified urine, unless *albumen* or *caseous matter* is present.

9. *Infusion of gall-nuts* or *tannin* precipitates at least two constituent principles, namely, *mucus* and the *extractive matter*, which last is also precipitated by acetate of lead.

10. *Red and blue litimus papers* are used to detect *free alkali* and *acid*.

11. *Yeast* is employed to discover the presence of *sugar* in urine, by exciting the vinous fermentation.

DISCRIMINATION (BY CHEMICAL TESTS) OF URINARY CALCULI CONSISTING OF A SINGLE DEPOSIT, OR OF ALTERNATING CALCULI.

1. *Bone-earth calculus*.—Insoluble in potash and in acetic acid, soluble in dilute nitric and hydrochloric acids. Before the blowpipe it first becomes black, and afterwards white: it is fused with difficulty.

2. *Ammonio-magnesian phosphate*.—It does not dissolve in potash, but evolves ammonia. Soluble in cold acetic and dilute nitric acids, and reprecipitated by ammonia. It gives off ammonia at 212° , and melts into a white pearl before the blowpipe.

3. *Fusible calculus*.—A portion is dissolved by acetic acid, and the remainder by hydrochloric acid. It readily fuses into a pearly bead before the blowpipe.

4. *Uric acid calculus*.—Readily soluble in potash, and is reprecipitated by acids. In strong nitric acid it dissolves with effervescence, the solution leaving, when evaporated to dryness, a residue, which on treating with an excess of ammonia becomes of a purple-red colour. Uric acid is nearly insoluble in hydrochloric acid. Before the blowpipe it evolves an ammoniacal odour and blackens, leaving a minute portion of a white ash, which possesses an alkaline reaction.

5. *Urate of ammonia calculus*.—It is soluble in potash with evolution of ammonia. Is readily soluble in alkaline carbonates, while uric acid is not. With nitric acid it behaves as uric acid does. It usually decrepitates before the blowpipe.

6. *Cystic oxide calculus*.—It is soluble in alkalies and in the carbonates of the fixed alkalies, giving a solution which is decomposed by heat, ammonia being first evolved, and after some time a combustible gas, smelling like bisulphuret of carbon. It is soluble in phosphoric, hydrochloric, sulphuric, nitric, and oxalic acids; and insoluble in water, alcohol, bicarbonate of ammonia, and tartaric, citric, and acetic acids. Before the blowpipe it exhales a peculiar foetid odour.

7. *Xanthic oxide calculus*. — It is completely dissolved by potash, and is reprecipitated by carbonic acid, white, becoming on drying, a pale yellow agglutinated mass, which possesses a waxy appearance. It is soluble in nitric acid with effervescence. When that solution is evaporated to dryness, and the residue treated with ammonia, no red colour is developed, as with uric acid. This calculus is very slightly soluble in hot water, and in hydrochloric and oxalic acids. Concentrated sulphuric acid dissolves it, forming a yellow solution.

8. *Oxalate of lime calculus*. — Insoluble in potash: it is decomposed by digestion in carbonate of potash, with formation of carbonate of lime and oxalate of potash. Insoluble in acetic, but soluble in hydrochloric and nitric acids. When heated to dull redness, it is converted into carbonate of lime, and then dissolves in acids with effervescence. Before the blowpipe, pure lime remains, which, when moistened, produces an alkaline reaction on test paper.

9. *Carbonate of lime calculus*. — It dissolves with effervescence in dilute acids, affording a solution which is precipitated by oxalate of ammonia.

10. *Calculi containing silica* leave, after calcination before the blowpipe, an infusible ash (principally silica), which dissolves in a melted bead of carbonate of soda with effervescence, producing a vitreous pearl, more or less limpid.

CHAPTER IV.

QUANTITATIVE ANALYSIS.

PRELIMINARY OBSERVATIONS.

THE portion of material employed in a quantitative analysis ought not to exceed thirty grains: from twenty to twenty-five grains of an inorganic body is the most convenient quantity, except in those cases in which a substance existing in minute proportion is to be determined, when more may be used. When a larger amount is operated on, the chances of error in the analysis are seldom reduced, while, from the greater quantities of precipitates to be filtered and washed, and of liquids to be evaporated, a much longer time is necessary for the execution of the analysis. If the substance to be analyzed is solid and pulverizable, it must be reduced to a fine powder, and cautiously dried, to get rid of all hygrometric moisture without expelling any of its chemically combined water. A temperature of from 100° to 150° Fahr. is quite sufficient to effect this; but if the body parts with its combined water at that degree of heat, it is then necessary to dry it at common temperatures, by exposure to a surface of oil of vitriol in a close vessel. The oil of vitriol is most conveniently contained in a shallow bason, across the top of which are placed wires to support a piece of paper, on which the substance to be dried is strewed. The bason is placed on a level surface, and covered with a bell jar, having a ground bottom; or, if necessary, under the receiver of an air-pump, in which a vacuum is maintained. Six or eight hours exposure is generally sufficient. To prevent the absorption of any water during weighing, the substance should be weighed in a tube about two inches in length and a quarter of an inch in diameter, transferred directly to the vessel in which it is to be dissolved, and the weight of the empty tube with its adhering particles ascertained and deducted from the entire

weight of the tube and substance. If the substance for analysis is a liquid, it may be weighed in a specific-gravity bottle, whose weight when empty has been previously ascertained; the liquid is poured out into a convenient vessel, and the bottle washed out several times with distilled water, the washings being added to the original liquid.

It has already been stated that a substance is generally obtained in a fit state for weighing by precipitation from a state of solution. Particular processes will now be described which are practised in the quantitative estimation of bodies, supposing those bodies or their compounds to exist in a state of purity, with the various methods of separating substances from one another in a state of mixture, for the purpose of their quantitative determination, commencing with the alkalies and earths, and proceeding to the metals proper and non-metallic bodies, those mixtures only being considered which occur in nature or are likely to be presented for examination. I have pursued as far as possible the plan adopted by Rose, in his valuable "*Handbuch der Analytischen Chemie*," of treating, under each substance, of the mode of separating it from those bodies which have been previously considered; so that a method of separating any two substances being required, the process, if given, will be found under the last of the two bodies in question.

In quantitative as in qualitative analysis, advantage is sometimes taken of certain similarities in the properties of bodies, which permit of their classification into groups, separable from each other by a single agent: thus sulphuretted hydrogen removes one class of metals proper from a solution, and hydrosulphate of ammonia another class, leaving earths and alkalies, the former of which may be precipitated by an alkaline carbonate.

SECTION I.

ALKALIES AND THEIR METALLIC BASES.

I. POTASSIUM.

If the potash to be weighed exists either as sulphate or nitrate, or as chloride of potassium, the salts are weighed as such, if pure, and the quantity of base contained in them is calculated.*

If in a state of solution as *neutral sulphate*, evaporate the liquid to dryness in a platinum capsule, or in one of porcelain if the solution contains anything which can act on platinum; transfer the residue carefully to a weighed platinum crucible, ignite with the cover on, to prevent loss by decrepitation, and weigh what remains. If an excess of sulphuric acid existed in the solution, the bisulphate of potash will be formed, which must be converted into the neutral sulphate before weighing. This is done by placing a piece of dry carbonate of ammonia in the crucible, when heated nearly to redness, and afterwards heating more strongly. The excess of sulphuric acid volatilizes as sulphate of ammonia, neutral sulphate of potash remaining.

When the potash exists as *nitrate*, the solution is evaporated to dryness in a weighed platinum capsule, and kept at a temperature of 212° until the weight becomes constant.

If as *chloride of potassium*, the only particular precaution to be observed is, that this salt must not be heated above a dull red heat, as it is volatile at somewhat higher temperatures.

If united to the weaker *volatile acids*, the salt of potash must be converted into the sulphate, or into chloride of potassium, by the addition of sulphuric or hydrochloric acid, evaporating to dryness and igniting with the precautions above mentioned.

When in combination with an acid which when free is soluble in alcohol, potash is sometimes estimated as the double chloride of platinum and potassium. The solution is eva-

* Tables for calculations in analysis are given in the Appendix.

porated to a small bulk, mixed with solution of chloride of platinum, and evaporated nearly to dryness. When treated with alcohol, the residue dissolves, with the exception of the double chloride of platinum and potassium, which is collected on a weighed filter (page 11.), washed with alcohol, dried and weighed on the filter. From the weight of the double salt that of the potassium it contains may be calculated.

II. SODIUM.

Soda may be weighed either in the state of sulphate of soda, or chloride of sodium; the same precautions being observed in the ignition of these salts as with the corresponding compounds of potassium.

Separation of potash from soda. — One process for the separation of these alkalies is founded on the solubility of the chloride of platinum and sodium, and the insolubility of the corresponding compound of chloride of potassium. Both alkalies are converted into chlorides, ignited and weighed. They are then redissolved in water and mixed with 3·7 times their weight of crystallized chloride of platinum and sodium, which precipitates the chloride of platinum and potassium. The precipitate is collected and weighed in the manner described under potassium, and from its amount, that of the chloride of potassium is calculated. The weight of the latter deducted from the weight of the mixed chlorides, gives that of the chloride of sodium.

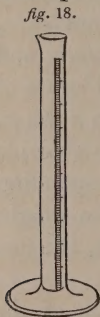
If hyperchloric (perchloric) acid is added to a mixture of potash and soda salts, it precipitates potash, but not soda. The mixture should be evaporated nearly to dryness, and the residue treated with alcohol, in which hyperchlorate of potash is quite insoluble. This salt is collected on a filter, dried and heated carefully to redness, when it leaves chloride of potassium, which may be weighed, and the corresponding amount of potassium or of potash calculated. The weight of the mixed salt of soda and potash being known, the loss expresses the quantity of soda.

When existing as sulphates (into which most salts of the alkalies may be converted by the addition of sulphuric acid),

add an excess of a solution of hyperchlorate of barytes: collect the precipitate, which consists of hyperchlorate of potash and sulphate of barytes, on a filter and wash it with hot water to dissolve out the former salt. If the sulphates did not contain excess of acid, the weight of the sulphate of barytes may be ascertained, and from it that of the sulphuric acid calculated. Evaporate the filtered liquid to dryness, and treat the residue with alcohol, by which hyperchlorate of soda and the excess of hyperchlorate of barytes are dissolved, and an insoluble residue of hyperchlorate of potash remains, which is filtered, washed with alcohol, dried, ignited, and weighed as chloride of potassium. The filtered alcoholic solution, containing hyperchlorates of soda and barytes, is evaporated to dryness, the salts are redissolved in water, and sulphuric acid is added to precipitate barytes. Filter and evaporate the solution, which now contains sulphate of soda, to dryness: ignite the residue with a little carbonate of ammonia, and weigh as neutral sulphate of soda. If the alkalies exist as chlorides, a similar process to the last is followed, hyperchlorate of silver being substituted for hyperchlorate of barytes, and hydrochloric acid for sulphuric acid. Chloride of silver is then formed in the steps in which sulphate of barytes would otherwise be obtained.

ALKALIMETRY.

The determination of the proportion of pure soda existing in a specimen of impure carbonate, as the *soda-salts* of commerce, is a process of considerable importance in connection with the arts. An exceedingly simple and easy method of ascertaining this has been devised, which consists in finding what quantity of an acid is required to destroy the alkaline reaction of a known weight of the specimen to be analyzed, which quantity of acid is of course equivalent to the amount of base present.



In this operation a tube measure, called an *alkalimeter* is employed (*fig. 18.*), about fourteen inches in height, and five-eighths of an inch in diameter, mounted

on a foot, and capable of holding, when full, a little more than 1000 grains of water. It is graduated up to this amount into one hundred divisions, numbered from the top downwards; each division, therefore, contains ten grains of water.

The acid employed is sulphuric, made of such a strength, that a single measure of the alkalimeter (ten grains measure of distilled water) is sufficient to neutralize exactly one grain of pure soda. Acid of this strength is obtained in the following manner: 170.6 grains of pure carbonate of soda (obtained by heating the bicarbonate to dull redness in a platinum or porcelain crucible) are dissolved in four or five ounces of hot water. This quantity contains exactly 100 grains of pure soda. A quantity, say a pint, of a dilute acid is prepared by mixing one part of oil of vitriol with ten of water by measure, with which the alkalimeter is filled up to 0 (1000 grains measure of water), and poured from this very gradually into the solution of carbonate of soda above mentioned, until it has destroyed the alkaline reaction of the latter on test paper, and the mixture has become very slightly acid. The quantity of acid which has been required to produce this effect is accurately observed. Supposing it to be 90 measures, this quantity is of course equivalent to 100 grains of soda; but an acid is wanted, of which 100 measures, instead of 90, are required to neutralize the soda, to procure which we have simply to add to 90 volumes of the acid, 10 volumes of water, so that the same amount of real acid which was before contained in 90 shall now be contained in 100 parts. For this, any vessel of sufficient capacity, divided accurately into 100 equal parts, will suffice. Such is the method of preparing the *test acid*, of which 1 alkalimeter measure (10 grains of water) is equivalent to 1 grain of pure soda: 22 measures should neutralize 100 grains of crystallized carbonate of soda, and $58\frac{1}{2}$ measures, 100 grains of anhydrous carbonate of soda.

In performing an actual analysis, 100 grains of the soda salt to be tested are weighed, dissolved in about three ounces of hot water, filtered if necessary, and neutralized with the

test acid poured from the alkalimeter. The number of measures used represents the amount of soda per cent. in the specimen. If, instead of 100 grains of the soda salt, 50 grains are operated on, the number of measures must of course be doubled to obtain the per-centage of soda.

The ordinary impurities in soda salts are chloride of sodium, sulphate of soda, and insoluble matters; but sometimes other bodies are present, which interfere with the above process, by neutralizing a portion of the acid; such as sulphuret of sodium, sulphite and hyposulphite of soda, which are liable to occasion a considerable error, by taking up the sulphuric acid. To avoid these sources of fallacy, it is recommended to mix the filtered solution of the salt with chlorate of potash, to evaporate to dryness, and to heat the residue to redness; by this, the sulphuret, sulphite and hyposulphite become converted into the sulphate of soda, which does not interfere in the process.

The analysis of a mixture of caustic soda and carbonate of soda is effected by first making an alkalimetical experiment to ascertain the whole amount of soda, and afterwards estimating the carbonic acid by a process which will be described under the analysis of carbonates. From the data thus obtained, the proportions of caustic soda and carbonate of soda are easily calculated. This may likewise be determined by means of a solution containing muriate of ammonia and chloride of calcium, from which carbonate of lime is precipitated proportional in quantity to that of the carbonate of soda in the alkaline mixture.

The same test acid used for soda may also be employed in analyzing the carbonates of potash; but as the equivalent of potash is higher than that of soda, the number of measures poured from the alkalimeter cannot represent the per-centage of this alkali, as it does of soda. A simple calculation, however, reduces the numbers for soda to those for potash; their respective equivalents being very nearly as 4 to 6, the number of measures necessary to saturate 100 grains of the specimen, multiplied by 6, and divided by 4, gives the per-centage of potash.

III. AMMONIA.

If the ammonia whose amount is to be estimated exists in aqueous solution, a slight excess of hydrochloric acid is added, and the solution is evaporated to dryness in a weighed platinum capsule, by means of a water-bath. When dry, the residue is weighed, again heated in the water-bath, and weighed. This is repeated so long as it suffers any loss. From the weight of the muriate of ammonia obtained, that of the ammonia is calculated. After the last weighing, the capsule is heated strongly, to sublime the muriate of ammonia. The loss now experienced, which should be pure muriate of ammonia, affords another datum in the calculation.

The estimation of ammonia in ammoniacal salts is performed in the following manner. A known weight of the salt is introduced into a small tubulated retort, having a tube funnel passing through a cork in the tubulure. The extremity of the funnel must reach nearly to the bottom of the retort, and be drawn out to a capillary just large enough to allow a liquid to flow through. The substance being introduced, the retort is connected with a receiver containing dilute hydrochloric acid, the beak of the retort just touching, but not dipping under, the surface of the liquid. A strong solution of potash is then introduced into the retort through the funnel, and heat is applied to distil the ammonia, which becomes free by the action of potash, into the receiver. If necessary, more solution of potash may be added, and afterwards water, to drive all the ammonia into the dilute acid. The solution of muriate of ammonia thus obtained in the receiver is evaporated in the manner above described.

IV. LITHIUM.

The best mode of estimating lithia, every base except potash and soda having been previously separated from the solution, is by means of the double phosphate of soda and lithia. Pure phosphate of soda and a little carbonate of soda are

added to the solution, and the whole is evaporated to dryness. On dissolving the dry mass in water, a white salt remains, insoluble in a solution of phosphate of soda, but slightly soluble in hot water. This is the double phosphate of soda and lithia. The quantity precipitated generally increases a little on standing. It is collected on a filter and washed with cold water. In consequence of the slight solubility of the double phosphate, the washing should not be prolonged beyond what is absolutely necessary. It should be heated to redness before being weighed.

If lithia is in combination with a volatile acid, and no other alkali is present, it may be estimated as *sulphate*, by simply adding sulphuric acid, evaporating to dryness, and heating to redness before weighing.

Separation of lithia from potash and soda.—When the three alkalies exist together in solution, first determine the entire weight of the mixed salts by evaporation to dryness; after weighing, redissolve the residue, and estimate the potash as chloride of platinum and potassium. Through the solution filtered from the double chloride, transmit a stream of sulphuretted hydrogen gas, to remove the platinum it contains from the excess of chloride employed, evaporate, and proceed to estimate lithia as the phosphate of lithia and soda. The sum of the weights of the potash and lithia salts (ascertained by calculation) deducted from the weight of the mixed salts first determined, leaves the weight of the soda salt.

SECTION II.

ALKALINE EARTHS AND EARTHS PROPER, AND THEIR METALLIC BASES.

I. BARIUM.

Barytes is weighed in the form of sulphate. On adding sulphuric acid to the solution containing barytes, the sulphate is immediately precipitated, but occasionally does not subside readily, and then passes through the pores of the filter. On heating the liquor, or on the addition of a little

nitric or hydrochloric acid, the precipitate aggregates, and may then be collected on a filter. The sulphate of barytes is washed, dried, and ignited in a platinum or porcelain crucible. From its weight that of barium or of barytes is calculated.

Barytes and alkalies are separated from each other by sulphuric acid; the liquid filtered from the precipitate of sulphate of barytes is evaporated to dryness, and the alkaline sulphates may be determined by one of the processes before described.

II. STRONTIUM.

Strontian is precipitated from its solutions for the purpose of weighing either as sulphate or as carbonate; but both of these salts possess a slight degree of solubility in water, and hence cannot be washed by water with impunity. If the strontian is united to an acid, which when free is soluble in alcohol, and nothing can be formed on the addition of sulphuric acid which is insoluble in weak alcohol besides sulphate of strontian, it is preferable to estimate the strontian as sulphate. The solution to be precipitated should be concentrated, mixed with alcohol and sulphuric acid, and the precipitate washed with weak alcohol, dried, ignited, and weighed.

To estimate strontian as carbonate, add to the solution an excess of carbonate of ammonia mixed with a little free ammonia, apply heat to the mixture, filter the precipitate, wash, dry, and heat it to redness. Carbonate of strontian does not part with carbonic acid at a red heat.

Strontian and barytes are separated from each other when in solution by hydrofluosilicic acid, which precipitates barytes (although incompletely), but not strontian. A more common, but less perfect method is, to convert the earths into dry chlorides of the metals, and then treat with alcohol, which dissolves chloride of strontium, but leaves chloride of barium undissolved.

Strontian is separated from the *alkalies* either as sulphate,

by sulphuric acid, or as carbonate, by carbonate of ammonia.

III. CALCIUM.

Lime is best precipitated from its solutions as oxalate, by means of oxalate of ammonia. The precipitate, after being washed and dried, is ignited at a red heat, when it becomes converted into carbonate of lime, which is weighed. In performing this process several precautions are necessary.

1. That the solution to be precipitated is either neutral or slightly ammoniacal, as oxalate of lime is soluble in acids. If the solution is acid, a slight excess of ammonia must be added; but this alkali may occasion the precipitation of a salt of lime, soluble in acids, but insoluble in water, and therefore precipitated on neutralization by an alkali; such, for instance, as the phosphate or arseniate. For such salts of lime the process by oxalate of ammonia cannot be employed. 2. As the oxalate of lime subsides very slowly, the mixture must be boiled for some time, and allowed to stand until all the oxalate has subsided, otherwise it is impossible to filter the solution perfectly. 3. In the conversion of oxalate into carbonate of lime by ignition, if a strong heat is employed, a portion of the carbonic acid of the carbonate may be expelled, thus subjecting the estimation of the lime to a considerable deficiency; to avoid this, after weighing the ignited carbonate, add to it a few drops of a strong solution of carbonate of ammonia, again ignite at a dull red heat, and weigh. Repeat this as long as any increase in weight is apparent.

In those salts which are soluble only in acids, and therefore precipitated from their solutions on the addition of ammonia, the lime is determined as sulphate in a manner precisely similar to that described under strontian.

Separation of lime from strontian. — When these two earths exist together in solution, their separation may be effected in the following manner. They are first converted into carbonates by the addition of carbonate of soda or potash; the precipitated carbonates are filtered, washed, and

dissolved in dilute nitric acid, to be converted into nitrates. The solution of the nitrates thus obtained is evaporated to dryness to expel the last traces of nitric acid, and the residue is treated with alcohol, which dissolves nitrate of lime, but leaves nitrate of strontian undissolved. Collect the nitrate of strontian on a filter, and wash it with alcohol. When washed, the salt may be dissolved in water, and the strontian estimated as sulphate; the lime of the nitrate in the filtered alcoholic liquid is estimated as oxalate.

Separation of lime from barytes. — On adding sulphuric acid to the solution containing these two earths, both sulphate of barytes and sulphate of lime are precipitated, but from the slight solubility of sulphate of lime in water, it may be completely separated from sulphate of barytes by a long continued washing on a filter. A solution of oxalate of ammonia is added to the filtered liquid, which should be concentrated by evaporation before the precipitated oxalate of lime is collected on a filter.

Lime is separated from the *alkalies* by oxalate of ammonia: on evaporating the liquid filtered from the precipitated oxalate of lime to dryness, and calcining the dry salt, the excess of oxalate of ammonia is expelled, together with any other ammoniacal salt which may be present, leaving a residue which may be treated in the usual way to obtain the alkali.

The method of analyzing limestones will be described under the Analysis of Carbonates.

IV. MAGNESIUM.

A solution of phosphate of soda, to which pure ammonia or its carbonate has been previously added, is employed to precipitate magnesia from its solutions for the purpose of weighing. The magnesia then falls in the state of phosphate of magnesia and ammonia. Several precautions are necessary in this operation. The solution should be concentrated to a small bulk by evaporation; it should be quite cold, and either neutral or slightly ammoniacal, as the precipitated phosphate of magnesia and ammonia is soluble in free acids.

Even carbonic acid, and, according to Stromeyer, alkaline carbonates, dissolve it to a small extent. If free carbonic acid exists in the solution, it must therefore be expelled by boiling; and if alkaline carbonates are present, they should be decomposed by the addition of hydrochloric acid: the solution is afterwards boiled, and the excess of acid neutralized by pure ammonia. Phosphate of magnesia and ammonia is also slightly soluble in pure water; hence, if the proportion of magnesia in the solution is small, no precipitate is at first produced on the addition of phosphate of soda with ammonia. The precipitate appears, however, on stirring for some time, which should never be omitted before filtration. Although soluble in pure water, phosphate of magnesia and ammonia is not in water which contains phosphate or muriate of ammonia in solution. A dilute solution of the latter salt is hence advantageously employed to wash the phosphate when collected on a filter. When dried and ignited, the muriate of ammonia is expelled, and the phosphate of magnesia and ammonia becomes reduced to phosphate of magnesia, $2 \text{Mg O} + \text{PO}_5$, containing 36.67 per cent. of magnesia, in which state this earth is weighed.

Magnesia may also be precipitated in the form of carbonate, by the fixed alkaline carbonates, but owing to the tendency of the latter to form soluble double salts with the carbonate of magnesia, this method of estimating magnesia is not more certain than the preceding. A solution of carbonate of soda (this salt being preferable to carbonate of potash) is added to the hot magnesian solution, and the liquid boiled until the precipitate is no longer bulky and light, but has become granular and heavy. As the presence of ammoniacal salts interferes with the precipitation of magnesia in the state of carbonate, these should be expelled from the solution by boiling with a sufficient quantity of carbonate of soda, until the odour of ammonia is no longer perceptible. The precipitate, if granular, and comparatively dense, is then collected on a filter, and the filtered liquid is evaporated to dryness. On treating the residue of the evaporation with boiling water, a minute quantity of carbonate of magnesia generally re-

mains undissolved, which should be added to that already on the filter. The precipitate, when washed, is dried, ignited strongly to expel carbonic acid, and weighed as pure magnesia.

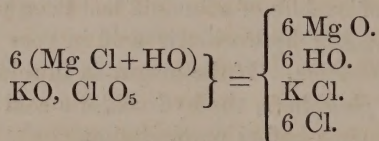
If magnesia exists in solution unmixed with other fixed bases, and not combined with a fixed acid, it may be estimated in the state of sulphate. The solution is evaporated to dryness, and if any ammoniacal salts are present, heated to redness in a platinum crucible. The residue is treated with dilute sulphuric acid, again heated to redness, and weighed when cold. If the magnesia already exists as sulphate, the addition of sulphuric acid is of course unnecessary.

The *separation of magnesia from lime* can be effected by oxalic acid. Ammonia in very slight excess is added to the solution of the two earths in hydrochloric acid, a previous addition having been made of muriate of ammonia, to prevent the precipitation of magnesia by ammonia. If the solution is very acid, a sufficient quantity of ammoniacal salt will be formed for the purpose, and the addition of muriate of ammonia is then unnecessary. If ammonia causes a precipitate of magnesia, this must be redissolved in hydrochloric acid, and a slight excess of the alkali again added, on which no precipitation will occur. The lime is then precipitated by oxalic acid, or by oxalate of ammonia, the oxalate of lime is collected on a filter, and magnesia is precipitated from the filtered solution as carbonate or ammonio-phosphate of magnesia. After the precipitation of lime as oxalate, the solution might deposit oxalate of magnesia on evaporation, if a large excess of oxalate of ammonia had been added: this is prevented by adding hydrochloric acid in very slight excess before the evaporation. If the magnesia in solution is to be precipitated as phosphate, the hydrochloric acid must be neutralized by ammonia before precipitation.

Döbereiner has lately proposed an excellent process for the separation of magnesia and lime, by the use of chlorate of potash. The solution of the two earths in hydrochloric acid is evaporated to dryness, and the residue is calcined in a platinum

crucible, to expel the excess of hydrochloric acid. Powdered chlorate of potash is then added so long as the evolution of chlorine is perceptible. On treating the dry mass with water, chloride of calcium and chloride of potassium are dissolved, and a residue of pure magnesia remains, which is filtered, washed, ignited, and weighed. The lime in the filtered solution may be precipitated by oxalate of ammonia.

When heated to redness alone, hydrated chloride of magnesium decomposes into magnesia and hydrochloric acid; hydrated chloride of calcium, however, resists decomposition in this manner, or, at least, at the temperature necessary for the decomposition of chloride of magnesium. Hence when a mixture of the two hydrated chlorides is ignited, on treating the calcined mass with water, chloride of calcium alone redissolves, if the ignition has been continued a sufficient length of time for the chloride of magnesium to be fully decomposed. Magnesia then remains as an insoluble residue. It is, however, with difficulty that this decomposition of the hydrated chloride of magnesium can be completely effected alone; but by the addition of chlorate of potash, as in Döbereiner's process for the separation of lime from magnesia, it is remarkably facilitated. This arises from the decomposition of the hydrochloric acid, as it is evolved from the hydrated chloride, by the oxygen of the chlorate of potash; water and chlorine being the products of the decomposition. Six equivalents of hydrated chloride of magnesium, and one of chlorate of potash, produce six equivalents of magnesia, six of water, one of chloride of potassium, and six of chlorine: or expressed in symbols:—



Separation of magnesia from the fixed alkalis.—One of the best means of effecting the separation of magnesia from the fixed alkalis is the following:—The magnesia is first precipitated by subphosphate of ammonia, and the precipi-

tated phosphate is filtered, washed, ignited, and weighed: acetate of lead is added to the filtered solution, to separate phosphoric acid (from the excess of phosphate of ammonia employed), and the precipitated phosphate of lead is separated by filtration. The lead in excess now existing in the solution is precipitated by supersaturation with sulphuretted hydrogen gas; the solution is filtered and evaporated to dryness. The residue of the evaporation consists of acetates of the alkalis, which may be converted into sulphates or chlorides by the addition of sulphuric or hydrochloric acid, and estimated by methods already described.

Liebig recommends a process for the separation of magnesia from the fixed alkalis, founded on the higher basic properties of barytes than magnesia. The solution containing magnesia and alkalis is mixed with muriate of ammonia, evaporated to dryness, calcined, and the residue is dissolved in a small quantity of water. To this solution, saturated barytes-water is added, and the magnesia hereby precipitated is collected on a filter: barytes is separated from the filtered liquid by sulphuric acid; the solution is filtered, evaporated to dryness, and the residue calcined with a little carbonate of ammonia (page 130.). Neutral alkaline sulphates remain, which may then be weighed.

Berzelius has recently recommended the following process for the separation of magnesia from the fixed alkalis, when existing in the state of chlorides, as is generally the case in mineral waters. The concentrated solution of the chlorides is mixed with protoxide of mercury (red oxide), and then evaporated to dryness. The chloride of magnesium and oxide of mercury undergo mutual decomposition, becoming magnesia and chloride of mercury; the latter combines with the alkaline chloride to form a double salt. The dry mass is treated with water, the solution is filtered, evaporated to dryness, and the residue calcined to expel all the chloride of mercury. What remains after the chloride of mercury is expelled, is the alkaline chloride, which may be weighed. The portion insoluble in water, consisting of a mixture of magnesia with the excess of oxide of mercury,

is ignited strongly to expel the latter. Pure magnesia then remains, in a fit state for weighing.

V. ALUMINUM.

For the purpose of weighing, alumina is precipitated from its solutions by ammonia or carbonate of ammonia. As pure ammonia exercises a slight solvent action on alumina, the carbonate should be used in all cases where it is admissible. When pure ammonia must be employed, however, the alumina is completely precipitated on boiling the solution for a short time. The precipitate is very bulky, and must be washed with hot water.

If alumina is to be precipitated from its solution in the caustic fixed alkalies, the alkali is first saturated with hydrochloric acid, of which a sufficient quantity is added to redissolve the alumina at first precipitated, and a solution of carbonate of ammonia, or ammonia, is then added to precipitate alumina.

Separation of alumina from magnesia, lime, and the fixed alkalies. — Supposing these bases to exist together in solution, their separation can be effected in the following manner. Muriate of ammonia is first added to the solution, to prevent the precipitation of magnesia by ammonia, which alkali is next added to precipitate *alumina*. If the solution was previously very acid, the addition of muriate of ammonia is unnecessary, as a sufficient quantity of an ammoniacal salt will be formed for the purpose, by the neutralization with ammonia. The ammonia should be perfectly free from carbonate, otherwise carbonate of lime will be formed. The precipitate of alumina should be filtered as rapidly as possible, to prevent absorption of carbonic acid from the atmosphere by ammonia, and washed with distilled water. To the liquid filtered from the precipitate of alumina, oxalate of ammonia is added to precipitate oxalate of *lime*, which is collected on a filter, washed, and converted into carbonate by ignition in a platinum crucible (page 138.). The precipitate of alumina produced by ammonia, carries down a little magnesia with

it, whatever amount of muriate of ammonia may exist in the liquid: this magnesia is separated by dissolving the alumina in caustic potash, magnesia remaining undissolved by the alkali. To perform this, the precipitate of alumina, while still moist, should be first dissolved in hydrochloric acid, an excess of caustic potash is then added to the solution, and the mixture heated in a porcelain or platinum capsule. The alumina, at first precipitated by the potash, redissolves in the excess of alkali, leaving magnesia undissolved, which is collected on a filter and washed. To the alkaline solution of alumina, hydrochloric acid is added until the precipitate of alumina at first produced is entirely redissolved; a solution of carbonate of ammonia in excess is then added to throw down the alumina. The small quantity of magnesia on the filter should be dissolved in hydrochloric acid, and this solution added to that filtered from the oxalate of lime, which contains the greater part of the magnesia, together with the alkalis. Processes for the separation of magnesia from alkalis have already been described.

When it is required to separate alumina from magnesia only, this may be accomplished by the same means as in the process just described, where lime and alkalis also are supposed to be present. Muriate of ammonia is first added to the solution, and then caustic ammonia. The small quantity of magnesia precipitated with the alumina is separated by hydrochloric acid and potash, in the manner described. The magnesia undissolved by potash is dissolved in hydrochloric acid, and added to the solution filtered from the precipitate by ammonia. From this solution magnesia may be precipitated, as the ammonio-phosphate (page 139.).

To separate alumina from lime only, the former is precipitated from the solution containing these earths by ammonia, which must be quite free from carbonate of ammonia, otherwise carbonate of lime will also be precipitated. The alumina should be collected on a filter as rapidly as possible, to prevent absorption of carbonic acid from the atmosphere by ammonia. After the filtered ammoniacal solution has been boiled to expel most of the excess of ammonia, the lime it

contains may be precipitated by oxalate of ammonia or oxalic acid.

From *barytes*, alumina is separated by precipitating the former earth as sulphate, by sulphuric acid or a soluble sulphate.

To separate alumina and *strontian*, the former may be precipitated by ammonia, the access of air being avoided as much as possible, to prevent the formation of carbonate of strontian.

Alumina and the *fixed alkalies* may be separated by adding carbonate of ammonia, or free ammonia, to the neutral or acid solution. On evaporating the solution filtered from the precipitate of alumina to dryness, and calcining the residue, the ammoniacal salts which are present volatilize, leaving the salts of the fixed alkalies.

Solution of aluminous minerals.—The solution of a few minerals, which consist entirely or chiefly of alumina, is very difficult to effect by ordinary means. M. Rose has shown that when corundum and aluminates* are reduced to a fine powder, they may be completely dissolved by first fusing them with an excess of bisulphate of potash, and then digesting the fused mass in water. The mineral should be pounded in a steel crushing-mortar, and not in an agate mortar, that no silica may be acquired by abrasion of the latter: it should be reduced to a further state of division by sifting through very fine muslin. A platinum crucible may be employed for the fusion with bisulphate of potash. From the solution of the fused salt in water, alumina may be precipitated by ammonia; but as a subsulphate of alumina is precipitated, instead of pure alumina, the precipitate should be separated by filtration, dissolved in hydrochloric acid, and again precipitated by ammonia. It is then pure alumina, which may be washed, ignited, and weighed.

* The minerals in which alumina acts the part of an acid are called *aluminates*.

VI. YTTRIUM.

Potash and ammonia are both employed to precipitate yttria from its solutions, in the quantitative determination of that earth. Hydrate of yttria falls, which may be collected on a filter, washed, ignited, and weighed as pure yttria. Except when yttria is in the state of chloride, potash is a better precipitant to employ than ammonia, as the latter alkali precipitates subsalts of yttria from the sulphate and nitrate.

The insolubility of yttria in potash affords the means of separating it from *alumina*, and its being precipitated by ammonia, although in the presence of an ammoniacal salt, from *magnesia*, *lime*, *barytes*, *strontian* and the *alkalies*.

VII. THORINUM.

Thorina is separated from most other bodies by precipitation as double sulphate of thorina and potash. A boiling saturated solution of sulphate of potash is added to the solution containing thorina, the mixture is allowed to cool, and the precipitate is filtered and washed with a cold saturated solution of sulphate of potash, in which the double salt is quite insoluble. After washing with solution of sulphate of potash, boiling water is poured on the filter, to dissolve the salt, the thorina of which is then precipitated by pure potash.

Thorina is separated from *alumina* by its insolubility in potash; and from *magnesia* and *lime* by being precipitated by ammonia in the presence of an ammoniacal salt. It is separated from alkalies by ammonia, which completely precipitates thorina.

VIII. GLUCINUM.

Ammonia is the reagent employed to precipitate glucina from its solutions. A voluminous precipitate of glucina falls, which contracts considerably on drying.

Glucina is separated from *thorina* and *yttria* by its solubility in potash, and from *alumina* by its solubility in car-

bonate of ammonia. To separate glucina from alumina, add a large excess of a saturated solution of carbonate of ammonia to the mixed solutions of the two earths, and allow the whole to stand for some time, with occasional agitation. When the precipitate of alumina no longer appears to decrease in bulk, collect it on a filter, wash, ignite, and weigh it, and evaporate the filtered solution to dryness. As the carbonate of ammonia volatilizes, the glucina is deposited: when the dry mass is heated to redness, to expel ammoniacal salts, it leaves pure glucina, the weight of which may then be ascertained.

A process has been recently recommended for the separation of alumina and glucina, which is said to be preferable to the preceding. Both glucina and alumina are soluble in a concentrated solution of potash; but if the alkaline solution is diluted considerably with water, and boiled, all the glucina is precipitated, while the alumina still remains in solution. The process is the following. Both earths are dissolved, with the assistance of heat, in a concentrated solution of potash; the solution is cooled and diluted with water. The precipitate which falls (glucina) is filtered and washed, and the filtered solution is boiled to precipitate the remaining glucina. The alumina which remains dissolved may be estimated in the usual manner (page 144.).

Berzelius * supposes it probable that glucina precipitated by ebullition from a dilute solution of potash, contains potash in chemical combination, for it is insoluble in cold alkaline ley after this operation; but by washing, all the potash it may contain is removed, and it again becomes soluble in the cold ley.

Glucina may be separated from *magnesia* by a process similar to that described for the separation of alumina from magnesia. The only difference is, that while caustic ammonia or carbonate of ammonia, may be used indiscriminately to precipitate alumina, caustic ammonia only should be employed for glucina.

Glucina is separated from *lime*, *strontian*, *barytes*, and the

* *Rapport Annuel*, 1841, 2^e Année, p. 79. (Paris edition).

fixed alkalies, by methods the same as those by which alumina is separated from the same bodies.

IX. ZIRCONIUM.

Zirconia is best precipitated from its solutions by potash. Ammonia may also be employed, but this alkali sometimes precipitates subsalts instead of pure zirconia. The precipitate is washed, dried, ignited with care, and weighed. Zirconia may be precipitated by sulphate of potash in the same manner as thorina. For this purpose, it is best to add sulphate of potash *in crystals* to the solution containing zirconia. The precipitate which falls is a double sulphate of zirconia and potash. If all excess of acid present in the solution is neutralized with potash, zirconia may be completely precipitated by sulphate of potash. As the precipitate is partly soluble in pure water, it should be washed with water rendered alkaline by the addition of a little caustic ammonia. On boiling the double sulphate of zirconia and potash with a solution of pure potash, hydrate of zirconia remains, which may be washed, ignited, and weighed.

SECTION III.

METALS PROPER, NOT PRECIPITATED BY SULPHURETTED HYDROGEN FROM THEIR ACID SOLUTIONS.

I. CERIUM.

Potash is the best precipitant for the oxides of cerium, ammonia having a tendency to throw down subsalts instead of the pure oxides. If the solution previously contained *protoxide* of cerium, the precipitate of protoxide produced by potash becomes converted by calcination into the *peroxide*, from the weight of which the amount of protoxide originally present must be calculated.

The oxides of cerium are separated from *glucina* and *alumina* by the insolubility of the former in caustic potash, and from *magnesia* and *lime* by ammonia.

II. MANGANESE.

Carbonate of potash is the reagent usually employed to precipitate protoxide of manganese from its solutions. The solution should be boiling at the time of the precipitation. Carbonate of protoxide of manganese falls, which may be collected on a filter. When the solution filtered from the precipitated carbonate is evaporated to dryness, a little more carbonate generally remains undissolved when the dry mass is treated with hot water; this portion is to be added to that already on the filter.

As carbonate of manganese is slightly soluble in ammoniacal salts, the whole oxide of manganese is not precipitated, if these are present in the solution, until a sufficient quantity of carbonate of potash has been added to decompose all ammoniacal salts. To effect their complete decomposition, the liquid should be mixed with carbonate of potash and evaporated nearly to dryness, fresh additions of the alkaline carbonate being made so long as any odour of ammonia is perceived. By this means ammoniacal salts may be completely expelled from the solution.

After washing and drying, the carbonate of manganese is heated to redness in an open platinum crucible; it then parts with carbonic acid, and acquires oxygen from the air, being converted into the red oxide of manganese (manganoso-manganic oxide, $\text{Mn O} + \text{Mn}_2 \text{O}_3$), from the weight of which that of protoxide or deutoxide, originally present in the substance under examination, may be calculated.

Separation of protoxide of manganese from alumina.—Protoxide of manganese may be separated from alumina by boiling the solution with an excess of caustic potash: of the mixture of oxide of manganese and alumina at first precipitated, the latter alone dissolves in the excess of alkali. The alumina in solution may then be estimated in the usual manner (page 144.), and the oxide of manganese, insoluble in potash, may be dissolved in hydrochloric acid, and precipitated as carbonate.

Protoxide of manganese and alumina may also be separated by ammonia, muriate of ammonia having been first added to the solution, to prevent the precipitation of protoxide of manganese.

Separation of protoxide of manganese from magnesia and lime.—When the amount of manganese present is relatively small, the following process is recommended. A sufficient quantity of muriate of ammonia is first added to the solution (unless it contains a large excess of acid) to prevent precipitation by ammonia, which is next added, and then an excess of hydrosulphate of ammonia to precipitate sulphuret of manganese. The precipitated sulphuret is collected on a filter, washed with water containing a little hydrosulphate of ammonia, and dissolved in pure hydrochloric acid: sulphuretted hydrogen is thus developed, to expel which, the solution is heated until it becomes inodorous. The solution is now filtered, and the manganese precipitated by carbonate of potash. The solution filtered from the sulphuret of manganese, containing lime and magnesia, is slightly acidified by hydrochloric acid, boiled to expel sulphuretted hydrogen, and filtered. The filtered solution is then supersaturated with ammonia; first lime is precipitated as oxalate, by oxalate of ammonia or oxalic acid, and afterwards magnesia, as the ammonio-phosphate.

When protoxide of manganese constitutes the chief ingredient, the following process is preferable. Carbonates of manganese and of the two earths are precipitated by a fixed alkaline carbonate, the solution being boiling at the time of precipitation. The precipitate is ignited at a dull red heat, to convert the carbonate of manganese into the manganoso-manganic oxide, and is then treated with very dilute nitric acid. This acid slowly dissolves the lime and magnesia with effervescence, leaving the oxide of manganese unacted on, which may be collected on a filter, washed, again ignited, and weighed. Any trace likewise of manganese dissolved by the nitric acid can be precipitated by hydrosulphate of ammonia, after neutralizing the acid by caustic

ammonia. The lime and magnesia may be separated from each other by a process already described.

VALUATION OF PEROXIDE OF MANGANESE.

A simple and expeditious process for the determination of the commercial value of the higher oxides of manganese, is of some importance from the extensive applications of these oxides in the arts. The following details of a process of this kind are given in Prof. Graham's "Elements of Chemistry" page 536.

"The value of these oxides is exactly proportional to the quantity of chlorine which they produce when dissolved in hydrochloric acid, and the chlorine can be estimated by the quantity of protosulphate of iron which it oxidizes.

"Fifty grains of the powdered oxide of manganese to be examined are weighed out, and also any known quantity, not less than 317 grains, of the sulphate of iron (copperas) employed in chlorimetry.* The oxide of manganese is thrown into a flask containing an ounce and a half of strong hydrochloric acid, diluted with half an ounce of water, and a gentle heat applied. The sulphate of iron is gradually added in small quantities to the acid, so as to absorb the chlorine as it is evolved, and the addition of that salt continued till the liquid, after being heated, gives a blue precipitate with the red prussiate of potash, and has no smell of chlorine, which are indications that the protosulphate of iron is present in excess. By weighing what remains of the sulphate of iron, the quantity added is ascertained; say m grains. If the whole manganese were peroxide, it would require 317 grains of sulphate of iron, and that quantity would, therefore, indicate 100 per cent. of peroxide in the specimen; but if a portion of the manganese only is peroxide, it will consume a proportionally smaller quantity of the sulphate, which quantity will give the proportion of the peroxide, by the proportion, as $317 : 100 :: m : \text{per-centage required}$. The per-centage of peroxide of manganese is thus obtained by

* Clean crystals of the salt, bruised in a mortar, and dried by strong pressure between folds of cloth.

multiplying the number of grains of sulphate of iron peroxidized, by 0. 317."

III. IRON.

This metal is best weighed in the state of peroxide, as obtained by precipitation from its solution by ammonia, potash, or by alkaline carbonates. If not already existing in solution as peroxide, but as protoxide or black oxide, it must be converted into the peroxide by heating with a little nitric acid, until ruddy fumes of nitric oxide are no longer evolved and the black colour sometimes acquired by the liquid on the addition of nitric acid has disappeared. The amount of metal, protoxide, or black oxide, which was formerly contained in the substance analyzed, is calculated in such a case from the quantity of peroxide obtained. Ammonia is the best precipitant to employ, as the peroxide of iron precipitated by potash always carries down with it some portion of the alkali, which is not removed by washing without great difficulty; and alkaline carbonates retain a little peroxide in solution at common temperatures. As much of the peroxide as possible is transferred, when washed and dried, to a crucible for ignition, the filter with the remaining peroxide being carefully burned outside the crucible (as described at page 8.), to prevent the reduction of any portions of oxide to the state of protoxide or black oxide by the carbonaceous matter of the filter.

Iron may be separated from several other bodies by precipitation as sulphuret, by hydrosulphate of ammonia. If the solution contains any free acid, it must first be neutralized by ammonia, which alkali may be added in slight excess, as the precipitation of a small quantity of peroxide of iron does not interfere with the result. A sufficient quantity of hydrosulphate of ammonia is then added, the precipitate is filtered, washed with water containing a little hydrosulphate of ammonia, and dissolved in pure hydrochloric acid. The solution thus obtained is heated to expel sulphuretted hydrogen, a little nitric acid is added to peroxidize the iron, the solution

is filtered if necessary, and the peroxide of iron is precipitated by ammonia.

Separation of peroxide of iron from protoxide of manganese. — The perfect separation of these oxides is not accomplished without some difficulty. In the process generally preferred it is effected by means of a neutral succinate or benzoate of an alkali, which precipitates peroxide of iron as benzoate, in a neutral solution, but not protoxide of manganese. If any iron exists in solution as protoxide, it must first be converted into peroxide by gently heating with a little nitric acid or aqua regia.

If the solution is acid, it must be rendered neutral by ammonia, muriate of ammonia being previously added to prevent the precipitation of protoxide of manganese. A very slight excess of ammonia may be added, so that a trace of peroxide of iron remains undissolved when the solution is gently heated. Solution of crystallized succinate of soda, of neutral succinate of ammonia, or of benzoate of ammonia may be used as the precipitant. A very bulky precipitate of benzoate or succinate of peroxide of iron falls, which, after being filtered and washed, is decomposed by pouring caustic ammonia on it, while still on the filter. By this, a large portion of the benzoic or succinic acid is removed, in the state of a soluble benzoate or succinate of ammonia, peroxide of iron, or rather a subsalt of that oxide, remaining on the filter, which last is dried, ignited with exposure to air, and weighed as peroxide. The object in removing the organic acid by ammonia is to avoid the reduction of the oxide during ignition. From the solution filtered from the precipitated succinate or benzoate, protoxide of manganese may be precipitated by carbonate of soda.*

Separation of peroxide of iron from magnesia. — These bodies are separated by a process similar to that just described for the separation of peroxide of iron from protoxide of manganese. Instead of neutralizing any excess of acid in the

* For another method of separating peroxide of iron from protoxide of manganese, see a process for the separation of oxide of nickel from peroxide of iron.

solution carefully, as when manganese is present, an excess of ammonia may be added at once, having made a previous addition of muriate of ammonia, unless the quantity of free acid is considerable. The precipitate, which consists of peroxide of iron with merely a trace of magnesia, is filtered, redissolved in hydrochloric acid, and the solution thus obtained is rendered exactly neutral by ammonia. Peroxide of iron is then to be precipitated as succinate or benzoate, and the liquid filtered from this precipitate is added to that filtered from the precipitate by ammonia, which contains the greater part of the magnesia. From the mixed solutions magnesia may be precipitated as the ammonio-phosphate.

Peroxide of iron may be separated from *alumina* by adding an excess of caustic potash to the solution. The alumina contained in the precipitate redissolves, and may be separated, after filtration, by neutralizing the alkaline solution with hydrochloric acid and precipitating by carbonate of ammonia.

Peroxide of iron may be separated from *lime*, *strontian*, and *alkalies* by ammonia, and from *barytes* by precipitating the latter as sulphate. For the means of separating *peroxide* and *protoxide of iron* from each other, see a process for the separation of peroxide of iron and oxide of nickel.*

ANALYSIS OF CLAY IRONSTONE.

The ordinary constituents of this ore are,—carbonic acid, silica, protoxide and peroxide of iron, alumina, magnesia, lime, and protoxide of manganese. Its analysis may be effected in the following manner:—

The powdered mineral is dissolved by boiling in aqua regia, with effervescence of carbonic acid and separation of silica, with, perhaps, a little alumina, as an insoluble powder. These are estimated, if necessary, by processes described under the analysis of Carbonates and of Silicates. The solution, which contains all the bases, with the exception of a little alumina, is evaporated to dryness; the residue is redissolved in dilute hydrochloric acid, and the solution filtered. Unless

* See also Note to the mode of estimating phosphoric acid in soils.

the solution is very acid, muriate of ammonia is now added, and afterwards an excess of caustic ammonia, which precipitates peroxide of iron and alumina, with small quantities of protoxide of manganese and magnesia. From the solution filtered from this precipitate, *lime* is to be precipitated, as oxalate, by oxalate of ammonia or oxalic acid. The precipitate by ammonia being filtered and washed, is dissolved in a small quantity of hydrochloric acid. This solution is then boiled with excess of caustic potash, to redissolve the *alumina* at first precipitated by potash, and which is estimated by the usual method (page 144.). The portion insoluble in excess of potash is dissolved in pure hydrochloric acid, the solution is carefully neutralized with ammonia, and peroxide of *iron* precipitated as succinate or benzoate, with the precautions already described. The filtered liquid, containing small quantities of magnesia and protoxide of manganese, is mixed with that filtered from the oxalate of lime, and, from the mixture, *manganese* is precipitated as sulphuret by hydrosulphate of ammonia, and *magnesia* from the filtered solution, as ammonio-phosphate, by phosphate of soda with ammonia (pages 139. and 151.).

If the amount of iron only is required, the other constituents of the ore being neglected, add ammonia in excess to the solution in aqua regia; after washing, boil the precipitate in solution of caustic potash to dissolve alumina, redissolve the peroxide of iron in hydrochloric acid, and precipitate it again by ammonia: dry, ignite, and weigh.

IV. NICKEL.

Nickel is weighed best in the state of oxide. It is completely precipitated as oxide by potash from a hot solution, whether ammoniacal salts are present or not. The precipitate, after washing with hot water, may be ignited and weighed.

Nickel is separated from some substances by precipitation as sulphuret, by hydrosulphate of ammonia in a slightly ammoniacal solution; but sulphuret of nickel being slightly soluble

in that reagent, its complete precipitation is not easily effected. As small an excess as possible of hydrosulphate of ammonia should be employed; the mixture with that reagent should be diluted and exposed in an open vessel to a very gentle heat, that the excess of the hydrosulphate may be volatilized, or decomposed by the oxygen and carbonic acid of the atmosphere. When the liquid loses its brown colour, the whole of the sulphuret of nickel is precipitated, and may be collected on a filter and washed with water, to which a little hydrosulphate of ammonia has been added. The filter and sulphuret are transferred together to a porcelain capsule, and digested in nitric acid or aqua regia, until the separated sulphur has a pure yellow colour, or is dissolved by the acid; the liquid is then diluted, thrown on a filter, and oxide of nickel is precipitated by potash. The digestion of the sulphuret in nitric acid should not be prolonged, as organic matter from the filter may be dissolved, which will impede the subsequent precipitation of oxide of nickel by an alkali.

Separation of oxide of nickel from peroxide of iron. — These oxides may be separated by boiling the carbonate of barytes or carbonate of lime with a solution of the two oxides (chlorides) in hydrochloric acid. The perchloride of iron in solution is decomposed by the earthy carbonate, with precipitation of peroxide of iron, and the formation of a corresponding quantity of chloride of barium or chloride of calcium; while chloride of nickel, on the contrary, is not affected by the earthy carbonate. The latter is added to the solution of the two oxides in hydrochloric acid so long as effervescence of carbonic acid occurs. When a sufficient quantity has been added, the mixture is boiled for a short time, and filtered. The precipitate of peroxide of iron with the excess of earthy carbonate, is then dissolved in hydrochloric acid, and if carbonate of lime has been used ammonia is added to re-precipitate peroxide of iron, avoiding exposure to the air as much as possible, to prevent the absorption of carbonic acid and formation of carbonate of lime. If carbonate of barytes has been used instead of carbonate of lime, barytes

may first be separated from the solution in hydrochloric acid by sulphuric acid, and peroxide of iron afterwards from the solution filtered from the sulphate of barytes, by ammonia.

From the solution filtered from the precipitate of peroxide of iron and excess of earthy carbonate, oxide of nickel may be precipitated by potash, then washed, dried, ignited, and weighed.

A similar process is sometimes adopted to separate peroxide of iron from protoxide of manganese, the two oxides being dissolved in hydrochloric acid. Peroxide of iron is precipitated through decomposition of the perchloride of iron, but protochloride of manganese is not affected by the earthy carbonate.

The particular precautions necessary to be adopted in this process are two: 1. That neither sulphuric, phosphoric, arsenic, nor boracic acid is present in the solution; and 2. that all the iron exists in the state of peroxide, as salts of the protoxide of iron are not decomposed by carbonate of lime.

On the property possessed by salts of the protoxide of iron to resist decomposition by an earthy carbonate, is founded a process for the separation of peroxide from protoxide of iron, when existing together in solution. When boiled with carbonate of barytes, the salt of the peroxide only is decomposed; the solution filtered from the precipitated peroxide and the excess of carbonate of barytes is boiled with a little nitric acid, to convert the protoxide of iron into peroxide, which last is precipitated by ammonia after the barytes has been separated by sulphuric acid.

Peroxide of iron can also be separated from oxide of nickel by means of an alkaline benzoate or succinate, as protoxide of manganese and peroxide of iron are separated from each other (page 154.).

Separation of nickel from manganese. — These metals are separated by a difficult process, founded on the reducibility of chloride of nickel to the metallic state, when heated to redness, by hydrogen gas, while chloride of manganese resists decomposition under similar circumstances. The reduction may be

performed in the apparatus, of which a figure is given at page 160. Chloride of manganese may be separated from the metallic nickel by water.

Oxide of nickel is separated from *magnesia* and the *alkalies* by hydrosulphate of ammonia, observing the precautions before mentioned in the precipitation of sulphuret of nickel, and from *alumina* by dissolving the latter in excess of caustic potash.

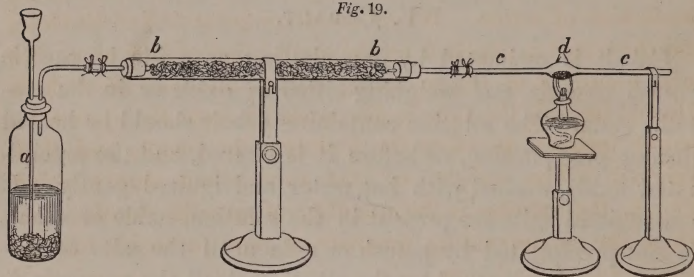
V. COBALT.

Cobalt is estimated by precipitating as oxide by caustic potash or soda, and weighing either as oxide or in the metallic state. The solution containing cobalt should be heated during precipitation, or before it is filtered, and the precipitated oxide washed with hot water and ignited gently. If ammoniacal salts are present in the solution, oxide of cobalt is not precipitated by potash or soda until the salts of ammonia are decomposed by the alkali, and all the ammonia is completely expelled by ebullition. But in such a case, it is better to precipitate cobalt as sulphuret by hydrosulphate of ammonia. To perform this, a slight excess of ammonia is first added to the solution, and then a sufficient quantity of hydrosulphate of ammonia to precipitate the cobalt completely. The precipitated sulphuret is collected on a filter and washed with water to which a little of the hydrosulphate has been added. When washed, the sulphuret is decomposed by digestion in nitric acid or aqua regia; the solution thus obtained is diluted, and oxide of cobalt precipitated by potash.

The ignition of protoxide of cobalt, previous to weighing, should be performed at a very gentle heat, as it acquires a little oxygen from the air when heated strongly in an open vessel, becoming converted into the peroxide. To avoid this source of error, the protoxide of cobalt may be collected in a weighed filter, placed when washed, together with the filter, in a platinum crucible, and heated in a water-bath so long as it suffers any loss in weight. (See description of the method of collecting, drying, and weighing a precipitate in a weighed filter, page 11.)

In cases where great accuracy is requisite, instead of weighing cobalt in the state of oxide, it should be weighed in the metallic state, the metal being obtained by reduction of the oxide by heating it strongly in an atmosphere of dry hydrogen gas. This reduction is performed in the apparatus depicted in the annexed figure: *a* is a bottle in which hydrogen gas is generated by the action of dilute sulphuric acid on iron or zinc; as the gas issues from the

Fig. 19.



bottle, it is dried by causing it to pass through the tube *bb*, which contains fragments of dry chloride of calcium. Connected with this chloride of calcium tube is the tube *cc*, having a bulb, *d*, blown in the middle, into which the oxide of cobalt for reduction is introduced. This tube should be of hard German glass.

Having ascertained the weight of the tube *cc* when empty, the whole of the oxide of cobalt may be introduced into the bulb. As it is difficult, however, to introduce the oxide without loss, it is better to take a part only, and to ascertain the proportion which the amount introduced bears to the entire amount by the three following weighings: —

1. The entire amount of oxide of cobalt after ignition.
2. The weight of the tube *c* when empty; and,
3. The weight of the tube *c* when the portion of oxide is introduced, from which that of the oxide can be estimated, by deducting the weight of the empty tube.

It may be calculated by the simple rule of proportion what quantity of metal is contained in the whole amount

of oxide, when it has been determined, by experiment, what is contained in the portion operated on.

The oxide being introduced, and the apparatus arranged as in the figure, hydrogen gas may be evolved by pouring concentrated sulphuric acid down the funnel of the bottle *a* on zinc and water already in the bottle. After the apparatus is filled with hydrogen, the bulb is heated to *redness* * by a spirit lamp. The cobalt is presently reduced from the state of oxide to that of metal, with evolution of heat and light, and production of water. When the decomposition is complete, and the water is expelled by the application of heat along the tube, a current of the gas being still maintained, the bulb is allowed to cool, the tube is disconnected from the apparatus, gently inclined to allow the hydrogen to escape, and then weighed. The weight of the tube when empty being known, the increase is the quantity of metallic cobalt, from which the corresponding quantity of pure protoxide may be obtained by calculation.

Separation of oxide of cobalt from oxide of nickel. — These oxides are not separated from each other without some difficulty. Mr. Phillips has described a process for the purpose, founded on the precipitability of oxide of nickel by potash from an ammoniacal solution of that oxide, while oxide of cobalt remains dissolved. To the solution of these oxides in an acid, a sufficient quantity of muriate of ammonia is added to prevent precipitation by caustic ammonia, which is then applied in excess. The mixture is largely diluted with water, which has been recently boiled to expel atmospheric air, and oxide of nickel is precipitated by potash, which alkali is added in sufficient quantity to decompose the ammoniacal salt. The precipitation should be performed in a vessel which can afterwards be closed air-tight. When the oxide of nickel has subsided, it may be collected on a filter, washed with hot water, and weighed; and the oxide of cobalt contained in the filtered solution, may be precipitated

* Oxide of cobalt may be reduced at a temperature much below redness, but the metallic cobalt thus obtained acts as a pyrophorus when it comes in contact with the air. It does not, however, if heated more strongly.

by hydrosulphate of ammonia. The necessity of avoiding exposure to the air, and of diluting the solution with water deprived of air, arises from the property which the ammoniacal solution of oxide of cobalt possesses, especially if strong, of absorbing oxygen, the protoxide of cobalt being converted into peroxide, which is insoluble in ammonia.

Professor Liebig has described very recently a method of separating cobalt and nickel by means of cyanide of potassium. When cyanide of potassium is added to a solution of a salt of nickel, a precipitate of cyanide of nickel is formed, which re-dissolves on adding an excess of cyanide of potassium, a double compound of cyanide of nickel and cyanide of potassium being formed, which is soluble in water. This double cyanide is decomposed by dilute sulphuric acid, with precipitation of cyanide of nickel.

When cyanide of potassium and an excess of hydrocyanic acid are added to any salt of cobalt, and the mixture warmed, there is formed the soluble double compound of percyanide of cobalt and cyanide of potassium (cobalti-cyanide of potassium), whose aqueous solution is not at all affected by boiling with hydrochloric, sulphuric, or nitric acid (Gmelin), unlike the analogous compound of cyanide of nickel and cyanide of potassium.

On adding hydrochloric acid in excess to a solution containing nickel and cobalt, with a little free acid, and gently heating, there are formed the double cyanide of nickel and potassium, and the double cyanide of cobalt and potassium. If to this mixture dilute sulphuric acid be added in the cold, three cases present themselves, according to the relative proportions which exist of nickel and cobalt.

(1.) " If the cobalt and nickel in solution are in the proportion of two equivalents of cobalt to three equivalents of nickel (quantities which correspond to their atomic proportions in the cobalti-cyanide of potassium), the precipitate produced is cobalti-cyanide of nickel, and is of a bluish-white colour.

(2.) " If the solution contains less nickel than corresponds to the above proportions " (as would be the case in

the analysis of an ore of cobalt), "there remains in solution a certain quantity of cobalti-cyanide of potassium, and the precipitate is still cobalti-cyanide of nickel.

(3.) "If there is more nickel present in the solution" (as in the analysis of an ore of nickel), "the precipitate is a mixture of cyanide of nickel and cobalti-cyanide of nickel."*

In the first and second cases, the precipitate produced by dilute sulphuric acid is boiled with the acid fluid until not a trace of hydrocyanic acid is observed to escape (or it may be evaporated nearly to dryness in a water-bath), and then slightly warmed with an excess of carbonate of potash or caustic potash; the cobalti-cyanide of nickel is thereby decomposed into pure oxide of nickel, or the carbonate, on the one hand, which is collected on a filter, washed, dried, and weighed; and an alkaline liquid, on the other hand, which contains the whole of the cobalt. The latter is evaporated to dryness, some nitrate of potash being added to it, and the residue ignited. On being afterwards treated with water, the oxide of cobalt remains behind, which may be weighed in the usual manner.

In the third case, which is that of the analysis of nickel ores, having the quantity of cobalt relatively small, it is necessary to add a considerable excess of muriatic acid to precipitate the cyanides dissolved in the cyanide of potassium, and to keep the mixture boiling at least one hour. The precipitate, as first produced, is a mixture of cyanide of nickel and cobalti-cyanide of nickel, but by boiling in hydrochloric acid, the cyanide of nickel is decomposed into chloride of nickel and hydrocyanic acid, the latter being expelled by the ebullition. The cobalti-cyanide of nickel is not affected by the acid. After the acid liquid has been boiled a considerable time, potash is added to the solution, as in the other cases. Chloride of nickel is then decomposed with precipitation of oxide, which falls completely if all the hydrocyanic acid has been expelled by ebullition previous to the addition of the alkali, but not otherwise, as cyanide of potassium would be formed, which is capable of retaining

* *Philosophical Magazine*, third series, vol. xx. p. 269.

nickel in solution. The cobalti-cyanide of nickel, insoluble in hydrochloric acid, is also decomposed by the potash, with formation of nickel and an alkaline solution of cobalt. The oxide of nickel is collected on a filter, washed, dried, and weighed; and oxide of cobalt is obtained from the alkaline solution, in the manner above described.

Peroxide of iron may be separated from oxide of cobalt by processes similar to those by which the same oxide is separated from oxide of nickel. The best method is by boiling carbonate of barytes in the solution of the two oxides in hydrochloric acid, when peroxide of iron is precipitated through decomposition of the perchloride of iron, while the chloride of cobalt is not affected by the earthy carbonate. The peroxide of iron may be dissolved out from the excess of carbonate of barytes by sulphuric acid, and again precipitated by ammonia. From the solution filtered from the peroxide of iron and excess of carbonate of barytes, oxide of cobalt may be precipitated as sulphuret by hydrosulphate of ammonia.

Oxide of cobalt is separated from *alumina* by potash, and from *magnesia*, *lime*, and the *alkalies* by hydrosulphate of ammonia.

VI. ZINC.

Carbonate of potash and hydrosulphate of ammonia are the reagents generally employed to precipitate zinc from its solutions, for the purpose of weighing; the zinc, in both cases, being afterwards weighed in the state of oxide. When carbonate of potash is used, a sufficient quantity should be added to decompose all ammoniacal salts which may be present, as these prevent the precipitation of subcarbonate of zinc. The mixture should be evaporated to dryness, the residue treated with hot water, boiled and filtered. If no ammoniacal salts are present, the solution may simply be boiled with carbonate of potash and filtered. On ignition, the subcarbonate of zinc loses carbonic acid, and becomes converted into the oxide.

In the form of sulphuret, zinc may be separated from

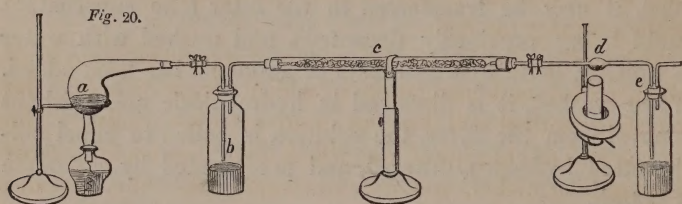
many other bodies by means of hydrosulphate of ammonia, in a neutral or ammoniacal solution. If the solution to be precipitated is acid, caustic ammonia should be added in sufficient quantity to re-dissolve the precipitate of oxide at first occasioned by the alkali. When hydrosulphate of ammonia has been applied, the sulphuret of zinc which is formed is allowed to subside before filtration, as it would pass through the pores of the paper if the solution were filtered immediately on being precipitated. When the sulphuret has subsided, it may be transferred to the filter (the supernatant liquid being previously decanted), and washed with water to which a little hydrosulphate of ammonia has been added. When washed, it is dissolved in hydrochloric acid without removal from the filter, the solution is boiled to expel sulphuretted hydrogen, filtered, and precipitated by carbonate of potash.

A precaution which should never be neglected in estimating zinc when precipitated as subcarbonate, is to examine whether the liquid filtered from the precipitate contains any more zinc in solution. This is best ascertained by hydrosulphate of ammonia; and the precipitated sulphuret, if any is formed, is converted into subcarbonate, and added to the quantity previously obtained.

Separation of oxide of zinc from oxides of cobalt, nickel, and manganese. — When the quantity of material operated on is very small (a few grains only), the oxides of cobalt, nickel, and manganese may be separated from oxide of zinc by means of caustic potash. On adding this alkali in excess to a solution of the oxides mentioned in hydrochloric acid, free from ammoniacal salts, oxide of zinc alone is re-dissolved by the excess, and it may be obtained from the alkaline liquid by neutralizing the latter with an acid, and precipitating by carbonate of potash. Although this method may be practised when the weight of the material does not exceed a few grains, it is impossible to separate the oxide of zinc from the other oxides completely by any excess of potash. When a large amount of the oxides is operated on, another method is therefore had recourse to. In that generally followed,

the chlorides of the metals are formed, and chloride of zinc, which is comparatively volatile, is separated from the other chlorides by distillation.

The first part of the process consists in ascertaining the united weights of the oxides by precipitation from their solution by carbonate of potash, and conversion into oxides by ignition. This being done, the whole or a known proportion of the oxides is then introduced into a bulb blown on a tube of hard German glass, *d* of the annexed figure, and weighed



with the tube. The apparatus being arranged as shown in the figure, the mixed oxides are exposed to the action of a stream of dry hydrochloric acid gas, at the highest temperature procurable by the Rose spirit lamp. The hydrochloric acid is evolved in the retort *a*, from a mixture of chloride of sodium and sulphuric acid; the gas is passed first into the bottle *b*, which contains a little concentrated sulphuric acid, next through the chloride of calcium tube *c*, and then over the oxides in the bulb. By being heated in hydrochloric acid gas, the oxides become converted into chlorides, and the volatile chloride of zinc distils. The exit limb of the bulb tube is bent at right angles about its middle, to enable it to dip into the receiver *f*, which contains water rendered slightly alkaline by ammonia, the surface of the liquid being immediately below the extremity of the tube. When no more chloride of zinc distils, the transmission of hydrochloric acid may be discontinued, the whole of the chloride of zinc which has condensed in the tube near the bulb being previously driven farther on by the application of heat. The part of the tube which contains this portion of chloride of zinc is divided from the bulb by means of a triangular file, and the

chloride is washed into the receiver, the solution in which should now contain all the zinc. From this solution, zinc may be precipitated by carbonate of potash, and weighed as oxide after being ignited.

The bulb containing the fixed chlorides is weighed, together with the divided portion of the tube, made clean and dry. The difference between the weight of the tube when empty and its present weight is the weight of the fixed chlorides in the bulb. After weighing, the bulb may be washed out with water, and the cobalt, nickel, and manganese estimated by the ordinary methods.

Although preferable to the process of separating zinc from cobalt, nickel, and manganese in which potash is used, the preceding method still does not afford accurate results. The last portions of chloride of zinc which distil over contain traces of chloride of nickel, and notwithstanding this, the chlorides remaining in the bulb are not entirely free from zinc.

Berzelius has recently made known the following process for separating zinc from cobalt and nickel, which he considers to be very exact. Having first separated the greater part of the oxide of zinc by boiling the mixed oxides in caustic potash, the residue is washed, first with cold and afterwards with boiling water, to withdraw the last traces of potash, ignited, and weighed. After weighing, the oxides are intimately mixed with finely pulverized sugar, which does not leave an incombustible ash on calcination, the mixture is introduced into a porcelain crucible, and the sugar is carbonized by careful heating. The porcelain crucible is covered, and placed in caustic magnesia contained in an earthen crucible, also covered, and exposed for one hour to the highest temperature of a wind furnace. By this operation the oxides are reduced, zinc is volatilized and completely expelled, and nickel and cobalt remain in the crucible in the state of carburets. The residue in the crucible is treated with nitric acid, to dissolve nickel and cobalt, and the solution is evaporated to dryness in a weighed platinum crucible: on heating the residue to redness, metallic oxides remain, from whose weight that of

the oxide of zinc is calculated, being the loss upon the former weight.*

Another method has been proposed by M. Ullgren. The mixed oxides of zinc, cobalt, and nickel are weighed, and heated to dull redness in an atmosphere of hydrogen, in the apparatus of which a figure is given, page 160. The oxides of cobalt and nickel are reduced to the metallic state by hydrogen with formation of water, but oxide of zinc remains unaltered. When no more water is produced, the bulb is allowed to cool, the current of hydrogen being still maintained. One of the extremities of the tube *c* is then sealed before the blowpipe, and the tube is filled with a solution of carbonate of ammonia: the open end is corked, and the tube is kept for twenty-four hours at a temperature near 100° Fahr. The oxide of zinc not reduced by the hydrogen dissolves completely in the carbonate of ammonia, from which it may be obtained by evaporating carefully to dryness and igniting the residue; the oxide of zinc remaining is then weighed. The cobalt and nickel are dissolved in nitric acid, and determined by a process already described, page 161.

Separation of oxide of zinc from peroxide of iron. — On adding an excess of ammonia to the solution containing these oxides, the oxide of zinc at first precipitated is re-dissolved, and a residue of peroxide of iron left. When the proportion of zinc is small compared with that of iron, this process may succeed; but if the zinc is in considerable quantity, no excess of ammonia will separate it completely from the peroxide of iron. In such a case, these oxides are best separated by means of an alkaline benzoate or succinate. (page 154.)

Oxide of zinc may be separated from *lime* by means of oxalate of ammonia in a strongly ammoniacal solution, and from *magnesia* and the *fixed alkalies* by hydrosulphate of ammonia. (page 164.)

* *Rapport Annuel* of Berzelius, 1841. (2^e Année, p. 81. Paris edition.)

VII. URANIUM.

Both the peroxide and protoxide of uranium are precipitated from their solutions by ammonia; but as the peroxide is precipitated more completely than the protoxide, it is convenient, when the latter is to be estimated, to convert it into peroxide (uranic acid) by heating with a little nitric acid, before precipitation by ammonia. If the precipitated peroxide, when collected on a filter, is washed with pure water, it passes through the pores of the paper, making the filtered liquid milky: this inconvenience may be avoided, to a great extent, by employing a dilute solution of muriate of ammonia, instead of water, in the washing. The precipitate, besides peroxide of uranium, contains ammonia and water, both of which, together with oxygen, it loses on calcination, being converted into the protoxide of uranium, from the weight of which, that of the peroxide, if the latter was originally present, may be calculated.

If the solution, besides peroxide of uranium, contains a considerable quantity of an earth, the latter is also precipitated by ammonia, in a state of combination with the peroxide, although the earth is one which is not precipitated by ammonia under ordinary circumstances, as lime, magnesia, and barytes. In such a case, when the precipitate is washed, it should be dissolved in hydrochloric acid, and the peroxide reprecipitated by ammonia; the precipitate which then falls is free from the earth, and may be washed with a solution of muriate of ammonia, dried, and calcined. By calcination it becomes the protoxide of uranium, which may be weighed.

Separation of uranium from iron. — On adding a large excess of carbonate of ammonia to a solution of the peroxides of these metals, the peroxide of uranium at first precipitated is re-dissolved in the excess, while peroxide of iron remains precipitated. If uranium and iron exist in solution as protoxides, the latter should be converted into peroxides by heating the solution with nitric acid before carbonate of ammonia is added.

VIII. CHROMIUM.

Estimation of chromic acid and oxide of chromium. — Chromic acid may be estimated by precipitation from a neutral solution by protonitrate of mercury; chromate of the suboxide of mercury is precipitated, which may be transferred to a filter and washed. When heated to redness, the precipitate leaves green oxide of chromium, from the weight of which that of the chromic acid may be calculated. According to Rose, chromic acid may be determined more accurately by reducing it to the state of oxide or chloride, by boiling with hydrochloric acid, by which chlorine is liberated. The reduction of chromic acid may be accelerated by the cautious addition of alcohol, any excess of which should afterwards be expelled by evaporation. Oxide of chromium is best precipitated by ammonia: the liquid is heated gently, filtered, and the precipitate ignited in a covered platinum crucible. By ignition, oxide of chromium is rendered insoluble in acids.

Separation of oxide of chromium from oxide of iron. Analysis of Chrome-iron. — The mineral being reduced to a fine powder, is mixed with about three times its weight of fused potash, with the addition of a small quantity of nitre, and the mixture is exposed to a dull red heat in a silver crucible. In this operation the oxide of chromium absorbs oxygen, and becomes converted into chromic acid, which unites with the alkali to form chromate of potash. The fused mass is digested in water until every thing soluble in that liquid is taken up, the alkaline solution is filtered, neutralized with acetic acid, and carbonate of ammonia is added to precipitate *alumina*. After the alumina has been gently ignited and weighed, it should be dissolved in hydrochloric acid to separate a little adhering *silica*, which remains undissolved, and may be collected on a filter, ignited, and weighed. The weight of the silica should be deducted from that of the alumina and silica previously weighed together. The chromic acid existing in the liquid filtered from the precipitate of

alumina is reduced to the state of *oxide of chromium* by boiling with hydrochloric acid, the oxide is precipitated by ammonia, filtered, dried, ignited, and weighed. The portion of the fused mass insoluble in water, consisting principally of *peroxide of iron*, is dissolved in hydrochloric acid, the solution is neutralized with ammonia, and peroxide of iron is precipitated by an alkaline succinate or benzoate in the manner described page 154. If magnesia exists in the ore, as sometimes occurs, it is contained in the liquid filtered from the succinate or benzoate of iron, from which it may be precipitated either as carbonate or ammonio-phosphate. (page 139.)

IX. TITANIUM.

Estimation of titanic acid. — This acid is best precipitated from its solution by ammonia, as small an excess as possible of that alkali being employed. It is recommended to expel the excess of ammonia, after precipitation, by exposing the liquid to a gentle heat in an open vessel. The precipitate is then transferred to a filter, dried, and ignited. Titanic acid should be weighed soon after ignition, as it rapidly absorbs moisture from the atmosphere.

Titanic acid may be separated from *oxides of iron, cobalt, zinc, and manganese*, in the following manner. A considerable quantity of tartaric acid is first added to the solution, which is then supersaturated with caustic ammonia; if sufficient tartaric acid has been previously added, no titanic acid is precipitated by the ammonia. By adding a slight excess of hydrosulphate of ammonia, all the other metals are completely precipitated as sulphurets, leaving in solution titanic acid, which may be obtained by filtering, evaporating the liquid to dryness, and igniting the residue in a platinum crucible until all combustible matter is destroyed. The residue is pure titanic acid, which may be weighed. From *lime* and the *fixed alkalis* titanic acid may be separated by ammonia.

SECTION IV.

METALS PROPER, PRECIPITATED BY SULPHURETTED HYDROGEN FROM THEIR ACID SOLUTIONS.

I. LEAD.

Estimation of lead, and modes of separating it from other metals. — Lead may be completely separated from all the metals which have been previously considered, by precipitation as sulphuret, by sulphuretted hydrogen, from a slightly acid solution. If the solution to be precipitated is not already acid, it is best rendered so by the addition of nitric acid, as hydrochloric, which is the acid generally used for that purpose with other metals, might cause a precipitate of chloride of lead, which is not decomposed by sulphuretted hydrogen with the same facility as a salt of lead in solution. When the proportion of lead is very small, solution of sulphuretted hydrogen in water may be employed; but in other cases it is better to pass the gas through the solution contained in a bottle, having a conical glass stopper. In all cases of precipitation by sulphuretted hydrogen, the gas must be transmitted, or the solution of the gas in water added, until the odour of the reagent becomes very perceptible, and contraction no longer occurs from absorption of the gas above the liquid, when the bottle containing it is agitated. Lead cannot be weighed in the form of the precipitated sulphuret, as it is almost impossible to obtain the latter without an admixture of free sulphur, and it cannot be ignited without decomposition. To obtain the lead in a fit state for weighing, it is necessary to convert the sulphuret into the sulphate of oxide of lead, which is done in the following manner. After being collected on a small filter* and washed, the sulphuret

* It sometimes happens that the sulphuret adheres so strongly to the sides of the vessel in which the precipitation was performed, and also to the tube through which the gas was passed, that it cannot be removed by washing out with water. When this is the case, having first washed out all the adhering liquid, the sulphuret of lead may be readily detached by pouring a few drops of fuming nitric acid on it. Sulphate of lead is then formed, which may be washed out and added to the other quantity of sulphate.

is dried and digested, while still in the filter, in a little fuming nitric acid, contained in a weighed platinum capsule, the acid being added very gradually to avoid loss by projection. By this, the sulphuret is entirely converted into sulphate, if the nitric acid is sufficiently strong; if such does not appear to be the case, from some sulphur having been set free without conversion into sulphuric acid, all the nitrate of lead then formed may be converted into sulphate by the addition of a few drops of sulphuric acid. The excess of acid is driven off by evaporation, and the capsule heated to bright redness to destroy the organic matter of the filter. In an open capsule the organic matter burns completely, without any reduction of the sulphate, which is then in a proper state for weighing. From the weight of the sulphate, that of metallic lead or oxide of lead originally present may be calculated.

Lead is also precipitated from its solutions, for estimation, as oxalate, by oxalate of ammonia, as sulphate by sulphuric acid, and as subcarbonate by carbonate of ammonia. In general, Rose gives the preference to oxalate of ammonia. As in the precipitation of oxalate of lime, it is necessary that the solution be neutral, or slightly ammoniacal. After washing and drying the precipitated oxalate, it is removed from the filter, which is burned separately, to avoid any reduction of the oxide by the combustible matter. The ash of the filter is added to the oxalate of lead, which is converted into oxide by ignition, and then weighed.

As caustic potash dissolves oxide of lead, it is sometimes used as the means of separating this oxide from those which are insoluble in potash; after being filtered, the alkaline solution should be saturated with acetic acid, and the lead precipitated by oxalate of ammonia.

From the facility with which compounds of lead are reduced to the metallic state, and an alloy with platinum formed, it is necessary to use porcelain crucibles in the ignition of all lead compounds, except the sulphate.

Analysis of chromate of lead. — The chromate being reduced to a fine powder, it is boiled in hydrochloric acid, with

the addition of a little alcohol, when it becomes converted into chloride of chromium, soluble, and chloride of lead, insoluble, in alcohol. The chloride of lead is collected on a weighed filter, washed with dilute alcohol, dried and weighed with the filter, as described at page 11. The alcohol being expelled from the filtered liquid by evaporation, the oxide of chromium is precipitated by ammonia. (See p. 170.)

II. CADMIUM.

Cadmium may be separated from all those metals which have been previously considered, with the exception of lead, by passing sulphuretted hydrogen gas into the solution previously rendered slightly acid. If the solution is neutral, hydrochloric acid may be employed to acidify it. The precipitated sulphuret is collected on a filter, washed, and transferred, while still moist and in the filter, into a capsule, and digested with the assistance of a gentle heat in concentrated hydrochloric acid. The sulphuret is thereby decomposed, being converted into chloride of cadmium, with evolution of sulphuretted hydrogen. When the conversion of sulphuret of cadmium into chloride is completed, and the odour of sulphuretted hydrogen no longer perceptible, the solution may be filtered, and cadmium precipitated from it as carbonate, by carbonate of potash. The carbonate is collected on a filter, washed, dried, and heated to redness before being weighed. Ignition reduces carbonate of cadmium to the state of oxide.

The filter must be burned separately, as described at page 8., when compounds of lead and some other metals are ignited, oxide of cadmium being easily reduced to the metallic state, and the metal lost from its volatility.

The insolubility of oxide of cadmium in caustic potash affords the means of separating it from *oxide of lead*. (See p. 173.)

III. BISMUTH.

By means of sulphuretted hydrogen, bismuth may be

separated from all bodies which are not precipitated by that reagent from their solutions in acids. The liquid through which sulphuretted hydrogen is passed should be diluted; but as the dilution of bismuth solutions by pure water causes the precipitation of subsalts of bismuth, a quantity of acetic acid should be added before dilution. The acetic acid completely prevents precipitation when the liquid is diluted. The solution may now be saturated with sulphuretted hydrogen, and the precipitated sulphuret be filtered, washed, and decomposed by digestion in strong nitric acid: the nitrate of bismuth is thus formed, and sulphur is set free. When the separated sulphur has a pure yellow colour, the sulphuret of bismuth is completely decomposed; the solution is then diluted (a sufficient quantity of free acid being present to prevent precipitation), filtered, the sulphur is washed with water slightly acidified by nitric acid, and oxide of bismuth is precipitated from the solution by carbonate of ammonia. Bismuth is completely precipitated from its solutions by carbonate of ammonia after standing for a few hours in an open vessel, although on first adding an excess of the carbonate, a considerable quantity of the precipitate may be re-dissolved. The precipitated subcarbonate is ignited in a porcelain crucible, and weighed as oxide. The filter should be burned separately to avoid reduction of the oxide.

When bismuth is to be separated from substances which are not precipitated by carbonate of ammonia, it is unnecessary to convert that metal into the sulphuret, but it may be precipitated at once by the alkaline carbonate. It is absolutely necessary, however, that the solution be free from hydrochloric acid, otherwise the precipitate of subcarbonate will be mixed with an oxichloride of bismuth, which resists decomposition by any excess whatever of the alkaline carbonate. In such a case, the sulphuret of bismuth should first be precipitated by sulphuretted hydrogen, and that decomposed by nitric acid in the manner above described.

Separation of bismuth from lead. — According to Liebig, bismuth may be separated from lead by carbonate of lime, which throws down bismuth, but not lead, from a cold solu-

tion of the nitrates of these metals. They may also be separated by adding to the solution in nitric acid, which must be free from hydrochloric acid, a sufficient excess of caustic potash to re-dissolve the oxide of lead at first precipitated. (See p. 173.) Oxide of bismuth remains undissolved.

Another method of separating bismuth from lead has been recently described by M. Ullgren. Carbonate of ammonia is added to the solution of the two metals to precipitate the subcarbonates, which are filtered and dissolved in acetic acid. A piece of perfectly pure lead is immersed in the solution, and the containing vessel is carefully closed and set aside for several hours. Metallic bismuth is then precipitated on the lead, an equivalent quantity of the latter being dissolved. When the precipitation is complete, the plate of lead may be separated from the bismuth, washed, dried, and weighed. The precipitated bismuth is collected on a filter, washed with distilled water which has been previously boiled to expel air, and dissolved in nitric acid. The solution of nitrate of bismuth is evaporated to dryness and calcined; pure oxide then remains, which may be weighed. To the solution of lead filtered from the precipitated bismuth, carbonate of ammonia is added, and the precipitate is washed, calcined, and weighed as oxide of lead. By the loss in weight sustained by the metallic lead employed in the process, it is known how much oxide of lead must be deducted from the preceding quantity.*

IV. COPPER.

Copper is precipitated from its solutions, for the purpose of estimation, in the state of oxide by caustic potash: the liquid should be rather dilute when precipitated, and always boiled before filtration. In the cold, potash in excess produces with salts of copper a bulky blue precipitate of hydrate of the protoxide, which, on the application of heat, becomes dark brown or black, very dense, and anhydrous. The precipitate is washed on a filter with hot water, dried,

* Berzelius, *Rapport Annuel*, 1841. (2^e Année, p. 83. Paris edition.)

and ignited with the usual precautions to prevent reduction by the combustible matter of the filter. Should a portion of oxide, however, be reduced to the metallic state, it is generally re-converted into oxide by the current of air produced by the combustion of the filter. A portion of the precipitated oxide of copper adheres so tenaciously to the bottom of the vessel in which it was boiled, as not to be removed to the filter without some pains by mechanical means: this, however, is easily effected by dissolving it in a few drops of hydrochloric acid, and re-precipitating the oxide by potash.

Copper is also precipitated for estimation in the metallic state by means of a piece of polished iron; but this method is not admissible in a delicate analysis.

As sulphuret, copper may be separated from many other metals by passing sulphuretted hydrogen gas through the acid solution. The precipitate should be filtered * rapidly, and washed with water impregnated with sulphuretted hydrogen; for when washed with pure water, or water holding air in solution, it oxidizes and dissolves to a small extent, and therefore passes through the filter. When clean, the sulphuret is transferred to a capsule, while still moist and on the filter, and digested in nitric acid or aqua regia, until entirely decomposed; the separated sulphur, if any remains undissolved by the acid, having a pure yellow colour. The solution is diluted, filtered, and decomposed by potash. By the prolonged action of nitric acid or aqua regia on the filter, a soluble organic compound is produced, whose presence impedes the precipitation of oxide of copper by potash, some of the oxide still remaining in solution after an excess of potash has been added. When this is the case (it is perceived by the blue colour of the solution), the portion of oxide must be again precipitated by sulphuretted hydrogen (as the action of this reagent is not affected by the organic matter), and the precipitated sulphuret treated as before. To avoid the inconvenience from the presence of organic matter, the conversion of the sulphuret

* The sulphuret which adheres tenaciously to the bottle in which the precipitation was effected, and to the tube which conducted the gas, may be detached by means of nitric acid. (See note, page 172.)

into nitrate should be effected as expeditiously as possible; or when the greater part of the sulphuret has been disengaged from the filter by the acid, the filter may be removed into another capsule, and the sulphuret remaining attached to it decomposed by a somewhat weaker acid.

Séparation of copper from lead. — M. Rose recommends the following method for the separation of these metals. Sulphuric acid is added to their solution in nitric acid, and the mixture is evaporated to dryness, so that the excess of acid may be expelled. On treating the dry mass with water, sulphate of lead remains undissolved, which is collected on a filter, dried, ignited, and weighed. From the filtered solution oxide of copper is precipitated by potash. The alkaline solution filtered from the oxide of copper contains a trace of oxide of lead, dissolved at first by the water as sulphate, and retained in solution afterwards by the excess of potash. In a delicate analysis, this small portion of oxide is worth separating, which is done by first neutralizing nearly the whole excess of alkali by an acid, and then precipitating oxide of lead as oxalate, by oxalate of ammonia.

Séparation of copper from zinc: analysis of brass. — Brass may be analyzed in the following manner. The alloy is dissolved in a small quantity of nitric acid, the solution is diluted and treated with sulphuretted hydrogen to precipitate sulphuret of copper, which is converted into oxide and weighed. From the liquid filtered from the sulphuret of copper, oxide of zinc may be precipitated, after boiling, by carbonate of potash. (page 164.)

Estimation of the suboxide of copper. — When it is required to estimate the suboxide of copper, the substance to be analyzed should be dissolved in nitric acid. That acid yields oxygen to the suboxide, converting it into the protoxide, which may be precipitated by potash in the usual manner. The amount of suboxide is then calculated from the weight obtained of protoxide.

V. SILVER.

Silver may be precipitated from its solutions, and weighed with great exactness, in the state of chloride, in which form it may be separated from all metals whose chlorides are soluble in water, or in dilute nitric or hydrochloric acids. The only metals from which silver cannot easily be separated in this form, are, mercury, when existing as suboxide, and lead, when the latter is in a very large proportion compared with the silver. Either hydrochloric acid, or the chlorides of potassium, sodium, or ammonium, may be employed to precipitate the chloride of silver, but preference is given to the first, as alkaline chlorides exert a slight solvent action on the precipitate. Before precipitation, the solution should be slightly acidified by nitric acid, and boiled for a few minutes previous to filtering, as the liquid would pass through the filter in a milky state without these precautions. Since chloride of silver is partially reduced by the agency of light, it is proper to keep it in the dark while being filtered: it answers very well to cover the funnel with a piece of blackened paper. When washed and dried, the chloride of silver is removed from the filter into a weighed porcelain crucible, and heated until fusion takes place. The filter is burned separately, its ash being received and calcined in a weighed platinum crucible cover, which is used, after calcining the ash, as the cover for the crucible in which the chloride is fused. From the weight of the chloride of silver, that of the metal or oxide may be calculated. After weighing, the fused chloride can be easily detached from the crucible, unless the inside of the latter is rough, by digestion in water for a day or two.

Estimation of silver in alloys.—An alloy of silver with other metals, may be analyzed by dissolving it in nitric acid, and precipitating chloride of silver from the solution by hydrochloric acid; or, if the alloy is insoluble in nitric acid, it may be treated with aqua regia, when chloride of silver is formed, while the other metals dissolve. A trace of chloride of silver may be dissolved by the acid, but it is precipitated

on dilution with water. The chloride of silver may be collected and weighed in the usual manner. From the solution filtered from the chloride of silver, the other metals may be obtained by processes described elsewhere.

Alloys of silver with metals which are oxidable by the air at high temperatures, such as *lead* and *copper*, are analyzed or *assayed* by the process of *cupellation*, which is briefly the following. It consists in fusing the alloy, mixed with a considerable quantity of pure metallic lead, in a small crucible formed of bone earth, which is called the *cupel*. By placing the cupel near an opening in the side of a furnace, air is caused to pass over the surface of the fused metals, which, with the exception of silver, become oxidized; the oxides formed, although by themselves infusible, dissolve in the fused oxide of lead, and are absorbed with it by the porous cupel, leaving a button of pure metallic silver, which may afterwards be detached and weighed. This operation is practised to a great extent where several such assays are required at the same time; but for a single analysis it is more convenient and accurate to dissolve the alloy in nitric acid, and precipitate the silver as chloride by dilute hydrochloric acid. When lead is present, the solution to be precipitated should be dilute, and the precipitate washed with hot water, for chloride of lead, though soluble in hot, is very slightly so in cold water, and might therefore be precipitated with the chloride of silver on adding hydrochloric acid. By long washing, however, with boiling water, the chloride of lead may be entirely removed.

VI. GOLD.

Gold may be separated from most other metals by precipitation in the metallic state from its solution by deoxidizing agents, such as protosulphate or protochloride of iron, proto-nitrate of mercury, oxalic or sulphurous acids. Protosulphate and protochloride of iron are the precipitants generally employed. On adding protochloride of iron to a solution of the chloride of gold, the latter yields all its chlorine to the former, which becomes perchloride of iron, metallic gold

being precipitated. A corresponding change occurs when the protosulphate of iron is employed. If a large excess of nitric acid is present in the liquid to be precipitated, it should be expelled by evaporation to dryness before adding the salt of iron; the residue is moistened with a little hydrochloric acid, to render it soluble in water, and also to prevent the subsequent deposition of a subsalt of the peroxide of iron on exposure to the air. Protosulphate or protochloride of iron being added, the mixture should be digested for a few hours at a gentle heat, and the precipitated gold filtered, ignited, and weighed.

When gold is to be separated from metals, the admixture of iron with which would render their determination difficult, oxalic acid, or an oxalate, is employed, instead of the proto-salt of iron. The excess of nitric acid in the liquid is first expelled by evaporation, and hydrochloric acid is added to redissolve the residue, and also to prevent the formation of any insoluble oxalates of the metals from which the gold is to be separated. The addition of oxalic acid causes an effervescence of carbonic acid and precipitation of metallic gold; the gold is not, however, completely precipitated without digestion at a moderate heat for a day or two.

The analysis of an alloy of gold and silver is one of frequent occurrence. If the proportion of silver is small compared with that of gold, a mass of the alloy is laminated, weighed, and heated with aqua regia, which dissolves the gold completely, leaving a residue of chloride of silver of the form of the alloy operated on. The chloride of silver should be disintegrated with a glass rod, the liquid considerably diluted with water (on which a little more chloride of silver is generally deposited), boiled, filtered, and the chloride of silver weighed. The excess of acid in the filtered liquid being expelled by evaporating to dryness, the residue is diluted, and gold is precipitated from it by oxalic acid. The solution filtered from the gold may then be examined for copper and iron.

If, however, the proportion of silver is large compared with that of gold, the coating of chloride of silver formed by the action of the aqua regia may effectually protect the

interior of the alloy from further action, although the latter is reduced to a very thin plate. In such a case, the alloy should be decomposed by boiling in strong nitric acid, the silver being then dissolved, and the gold separated in the form of a powder, which may be collected and weighed. If the alloy resists the action of hot nitric acid, it should be fused with thrice its weight of pure lead, such as that obtained by heating the acetate of lead to redness in a porcelain crucible over a spirit lamp. The lead alloy is readily acted on by nitric acid, all the metals being dissolved except gold, which remains in a pure state, and may be collected on a filter. From the filtered liquid, silver may be precipitated as chloride by a hot solution of chloride of lead.

VII. MERCURY.

The most convenient form in which mercury can be obtained fit for weighing, is in that of the metal itself, in which state it may also be separated from many other metals. The reducing agent generally employed to obtain metallic mercury from its compounds is protochloride of tin; the reduction of all mercurial salts, whether soluble or insoluble in water and acids (with the single exception of the sulphuret), being easily effected by means of that reagent. When, however, the admixture of tin with the bodies from which mercury is to be separated would render the subsequent determination of the latter difficult, instead of the protochloride of tin, phosphorous acid is employed, which becomes phosphoric acid, when brought into contact with a solution of either of the oxides of mercury, with precipitation of mercury in the metallic state.

If the mercury exists in solution, the routine of the operation is as follows. The liquid is first strongly acidified by hydrochloric acid, an excess of protochloride of tin which has been previously rendered clear by the addition of a few drops of hydrochloric acid is then added, and the mixture is boiled for a few minutes. The boiling should not be continued longer than a few minutes, to avoid the risk of losing

mercury by evaporation. When the precipitated mercury is completely deposited, the supernatant liquid should be decanted, and the precipitate boiled with concentrated hydrochloric acid, on which it generally loses its pulverulent appearance, and becomes converted into running globules. If the quantity of mercury is very small, it may be collected in a weighed filter, dried at a temperature not exceeding 120° or 130° Fahr., and weighed with the filter in a covered platinum crucible. (See page 11.) If the quantity of mercury, however, is not small, a better method of collecting it is the following. The precipitate having been boiled in hydrochloric acid (one object of which is to dissolve any peroxide of tin which may have been precipitated), the supernatant liquid is decanted, and the precipitate is washed by affusion of water acidulated with hydrochloric acid, and subsidence. When all the foreign bodies are removed by washing, the humid mercury is allowed to fall into a weighed porcelain capsule, and as much as possible of the liquid floating over the mercury is taken up by a piece of bibulous paper. The remaining moisture is expelled by evaporation at a temperature not exceeding 150° Fahr. When the mercury is dry, it may be weighed in the porcelain capsule.

If nitric acid exists in the solution from which mercury is to be precipitated, it must be previously expelled by boiling, with the occasional addition of hydrochloric acid, so long as the formation of any chlorine or nitrous acid is perceived from the simultaneous decomposition of nitric and hydrochloric acids. When the quantity of nitric acid is very considerable, it is better first to precipitate the mercury as sulphuret, by means of sulphuretted hydrogen, and then to convert the sulphuret into metallic mercury in the following manner:—The precipitate is collected in a small filter, and introduced while still moist and on the filter, into a flask containing about one ounce of pure and slightly diluted hydrochloric acid. Through this mixture, chlorine gas is passed, by which the sulphuret is completely decomposed, with formation of chloride of mercury (corrosive sublimate)

and free sulphur, some of the latter being slowly converted into sulphuric acid. When the undissolved sulphur is of a pure yellow colour, the transmission of chlorine may be discontinued; the liquid is then boiled to expel free chlorine, filtered, and metallic mercury is precipitated by protochloride of tin.

If the mercurial compound to be analyzed is insoluble, it is introduced, excepting in the case of the sulphurets, into a flask, treated first with strong hydrochloric acid, and then with an excess of a saturated solution of pure protochloride of tin. The mixture is boiled for a few minutes, and allowed to cool, the flask being well corked. When the solution is quite cold, and the decomposition complete, the supernatant liquid may be decanted, and the mercury washed and collected in a porcelain capsule, as described above.

Mercury in solution may be separated from many other bodies by sulphuretted hydrogen: if this reagent precipitates the pure *protosulphuret* (unmixed with free sulphur), the precipitate may be collected on a weighed filter, dried carefully, and weighed with the filter in a covered platinum crucible, the quantity of metal, protoxide or suboxide, being calculated from the weight of the protosulphuret. If sulphuretted hydrogen precipitates the *subsulphuret*, this compound, as well as the protosulphuret when mixed with free sulphur—as would occur, if nitric acid, a salt of the peroxide of iron, chromic acid, &c. existed in the solution—is decomposed by chlorine, in the manner before described, and the resulting chloride of mercury is reduced to metallic mercury, which is weighed. The reason why mercury cannot be well weighed in the state of subsulphuret, is, that a slight elevation of temperature causes the decomposition of the subsulphuret into protosulphuret and metallic mercury, and a portion of the latter might easily be lost, from its volatility.

Separation of the oxides of mercury from the oxide of silver.
— When mercury exists in the state of protoxide (red oxide), it may be separated from silver by first precipitating the latter from the solution, as chloride, by hydrochloric acid; and from the solution filtered from the chloride of silver, me-

tallic mercury is precipitated by protochloride of tin. This method cannot be practised when the mercury is in the state of suboxide (black oxide), as subchloride of mercury would then be precipitated with chloride of silver. In this case, it is necessary to convert the suboxide of mercury into protoxide, by gently heating with a little nitric acid; then to precipitate the silver as chloride; and, afterwards, if much nitric acid is present in the solution, mercury should be precipitated as sulphuret, which is converted into chloride by means of chlorine, in the manner before described.

Separation of protoxide of mercury from protoxide of copper.

— If these oxides exist in the dry state, and unmixed with other bodies, their proportions may be determined by expelling the oxide of mercury from a known weight of the mixture by heating to redness in a platinum crucible, and weighing the residuary oxide of copper. If in a state of solution, one method of effecting the separation is the following. The protosulphurets of both metals are precipitated by passing sulphuretted hydrogen gas through the solution, which should not contain free nitric acid, nor any substance capable of precipitating free sulphur. The sulphurets are collected in a weighed filter, washed rapidly, and dried in vacuo over sulphuric acid (page 128.). When dry and weighed, either the whole or a known proportion of the sulphurets is then introduced into a weighed tube retort with an elongated neck, and heated to redness: sulphuret of mercury is thereby volatilized, together with one half of the sulphur contained in the protosulphuret of copper: the latter, therefore, becomes reduced to the state of subsulphuret, which is weighed. Having calculated the quantity of protosulphuret of copper which corresponds to that of the subsulphuret weighed, the amount of sulphuret of mercury may be estimated as loss.

Estimation of mercury in amalgams. — The amount of mercury contained in an amalgam, that is, in an alloy of mercury with other metals, may be determined by ascertaining what loss in weight occurs on volatilizing the mercury by heating the amalgam to redness. If the other metals are

not oxidable by the air at high temperatures, a small porcelain crucible may be employed in this operation; but if the metals are oxidable, a tube retort should be used, the neck of which is elongated after the material is introduced, and sealed after the tube has been heated to redness.

By means of sulphuretted hydrogen, mercury in solution may be separated from the metals proper not precipitated by this reagent in their acid solutions, from earths and from alkalies. The precipitated sulphuret of mercury is converted into the chloride by means of chlorine, as described page 183., from which mercury is precipitated in the metallic state by protochloride of tin.

VIII. PLATINUM, PALLADIUM, OSMIUM, IRIDIUM AND RHODIUM.

Analysis of platinum ore. — The modes by which the metals accompanying native platinum are estimated and separated from each other, are well exhibited in the ordinary process for analyzing platinum ore.

Besides the metals peculiar to the ore, namely, platinum, palladium, osmium, iridium and rhodium, it contains small quantities of iron and copper. For the method described we are indebted to Berzelius: although an exceedingly complicated process, it is, I believe, the best we possess.

When every thing which seems to be foreign to the ore has been separated by mechanical means, it is digested in dilute hydrochloric acid, in order to remove the metallic iron and peroxide of iron with which the ore is generally mixed; it is then washed, carefully dried, and about thirty grains are weighed out for analysis. After being weighed, the ore is ignited, and dissolved in aqua regia with the assistance of heat, in a glass retort, furnished with a proper receiver which should be kept constantly cold. When the acid has been distilled so far that the liquid remaining in the retort possesses the consistence of a thick syrup, the mass is dissolved in a very small quantity of water, and the solution carefully poured off from the undissolved residue. The distilled acid, which is yellow, and contains some of the ore

projected by the effervescence, is returned to the residue in the retort and redistilled. If the liquid in the receiver is not then obtained in a colourless state, it must again be returned to the retort and redistilled.

(A.) When procured colourless, the distilled liquid is diluted with water, and ammonia is added to it, so that a slight excess only of free acid remains. To this solution, sulphuretted hydrogen water is added, and the mixture is set aside, out of the contact of air, until the precipitate produced by sulphuretted hydrogen, which is sulphuret of osmium, has completely subsided. The clear supernatant liquid is drawn off with a syphon, and the sulphuret collected on a weighed filter, washed, dried and weighed. According to Berzelius, this sulphuret contains between 50 and 52 per cent. of osmium.

(B.) If the saline mass in the retort gives a solution smelling of free chlorine when dissolved in water, that gas proceeds from the decomposition of chloride of palladium, and must be expelled by boiling. Should a precipitate appear, it is oxide of palladium; this is redissolved by hydrochloric acid, and the liquid is passed through a weighed filter, to separate some grains of a combination of osmium and iridium, together with some sand which could not be separated before the analysis. When dried, these matters are weighed on the filter without being ignited. If aqua regia containing too much nitric acid is employed to dissolve the ore, peroxide of iridium is occasionally precipitated, which passes through the filter, having the appearance of charcoal.

The filtered liquid is mixed, first, with twice its bulk of alcohol of sp. gr. 0.833, and afterwards with a concentrated aqueous solution of chloride of potassium, which must be added so long as a precipitate appears on agitation.

The precipitate is essentially a mixture of chloride of platinum and potassium, and chloride of iridium and potassium; but it contains, besides these compounds, small quantities of the corresponding double salts of rhodium and palladium. It is collected on a filter, and washed with alcohol of sp. gr. 0.896, to which a little chloride of potassium has been added,

till the filtered liquid gives no precipitate with sulphuretted hydrogen water.

The subsequent operations are now divided into two series: first, the treatment of the precipitate; and, secondly, the treatment of the alcoholic solution.

(C.) As much as possible of the double chlorides on the filter, when washed and dried, is removed, and carefully mixed with an equal weight of carbonate of soda: the filter, with what remains of the precipitate, being burnt, the ash is mixed with carbonate of soda, and added to the other mixture, and the whole is gently heated in a *porcelain* crucible until it becomes black throughout. The double chlorides are thus decomposed; the chloride of platinum is reduced to the metallic state, while the iridium and rhodium are oxidized. The saline mass is treated with water, by which the greater part of it is dissolved, and hydrochloric acid is poured on the residue to extract the alkali combined with oxides of iridium and rhodium.

The residue of the action of hydrochloric acid, consisting of metallic platinum and peroxides of iridium and rhodium, is washed, dried, ignited (the filter being burned separately), and weighed. This being done, it is fused in a covered platinum crucible with five or six times its weight of bisulphate of potash, in which the rhodium dissolves, and communicates to it a red colour. This operation is repeated with fresh bisulphate, until the salt no longer acquires a red or pink colour.

(D.) The quantity of *rhodium* may be determined in two ways: if the platinum and peroxide of iridium which remain undissolved after fusion with bisulphate of potash are washed, ignited, and weighed, the loss on the weight of the mixture of platinum and peroxides of iridium and rhodium before the fusion indicates *peroxide of rhodium*, which contains 71 per cent. of metal. Or, instead of this method, the saline solution of rhodium may be mixed with an excess of carbonate of soda, evaporated to dryness, and ignited in a platinum crucible: on re-solution in water, peroxide of rhodium remains, which, when washed and dried, is reduced

to the metallic state by heating in a tube in a stream of dry hydrogen gas. For the reduction, the apparatus of which a figure is given at page 160. may be employed. The rhodium thus obtained, sometimes contains palladium, which must be extracted by digestion in aqua regia; the acid in the solution is afterwards neutralized by an alkali; and palladium is precipitated as cyanide by cyanide of mercury. On being heated to redness, cyanide of palladium is decomposed into cyanogen gas and metallic palladium, which may be weighed. The weight of this portion of palladium is, of course, deducted from the rhodium.

(E.) The next operation is to obtain the amount of *iridium*. The metallic mass which remains after the separation of rhodium by bisulphate of potash is digested with very dilute aqua regia, to remove the greater part of the platinum it contains. The solution thus obtained appears very dark, from the presence of some suspended peroxide of iridium; but when the solid matter has subsided, it is of a pure yellow colour, and should then be decanted. The insoluble residue is treated with a mixture of concentrated aqua regia and chloride of sodium, and evaporated to dryness. The use of chloride of sodium is to prevent the formation of protochloride of platinum. When the dry mass is digested in water, peroxide of iridium remains as an insoluble residue, which is filtered and washed, first with a solution of chloride of sodium, and afterwards with muriate of ammonia to remove the common salt. If washed with pure water, the peroxide of iridium would pass through the pores of the filter. It is now burned with the filter, and to be obtained in a fit form for weighing, should be reduced to the metallic state by hydrogen gas, which may be done in the apparatus figured at page 160. A small quantity of iridium exists in the solution of chloride of sodium, to obtain which it is mixed with carbonate of soda, evaporated to dryness, and ignited. The product, when freed from soda salts by water, and from platinum by aqua regia, leaves peroxide of iridium, which is reduced to the metallic state, and added to that already obtained.

(F.) Having by these means obtained the weights of the rhodium and iridium, that of the platinum may be calculated. From the united weights of platinum, and peroxides of rhodium and iridium (C.); subtract the peroxide of rhodium (D.), and the iridium (E.), (the latter being increased 12 per cent. to convert it into peroxide), the remainder is the weight of the metallic platinum.

Such is one series of operations in this complicated analysis; there still remains for examination the alcoholic liquid filtered from the double chlorides.

(G.) This liquid is introduced into a bottle or flask, which will permit of being closed air-tight, and a current of sulphuretted hydrogen gas is passed through it to saturation. The bottle or flask is then closed, allowed to stand in a warm place for twelve hours, and if the precipitate has subsided, the liquid is filtered, and the alcohol evaporated off. During evaporation, an additional precipitate falls, which is to be added to the former. The precipitate by sulphuretted hydrogen consists of the sulphurets of palladium, rhodium, iridium and copper; and the liquid filtered from the precipitated sulphurets contains iron, rhodium and iridium, together with a trace of manganese. During the evaporation of the alcohol, a greasy sulphuret is deposited, which cannot be detached from the sides of the vessel by simple washing, but may be easily removed after all the adhering liquid has been washed away, by dissolving it in ammonia. The ammoniacal solution is evaporated to dryness in a platinum crucible, the residue is mixed with the other sulphurets, and the whole is roasted by heating, with exposure to the air, so long as sulphurous acid is produced. When completely oxidated, the mass is treated with muriatic acid, by which the subsulphates of copper and palladium are dissolved, while oxides of iridium and rhodium, with a trace of platinum, remain undissolved.

(H.) To the solution in muriatic acid of the roasted sulphurets of the last operation, a mixture of nitric acid and chloride of potassium is added. On evaporating the mixture to dryness, a dark-coloured saline mass is obtained, which

consists of chloride of potassium, chloride of copper and potassium, and chloride of palladium and potassium, of which the two first are separated from the third by treatment with alcohol of sp. gr. 0.833, in which the former dissolve, leaving the double salt of palladium as an insoluble residue, which is collected on a filter and washed with alcohol. When dried and ignited, this salt may be weighed: it contains 28.84 per cent. of palladium. The alcoholic solution filtered from the palladium salt is concentrated by evaporation, and oxide of copper precipitated from it by potash. (page 176.)

(I.) The portion of the roasted sulphurets (G.) insoluble in hydrochloric acid, consisting of oxides of iridium and rhodium, with a trace of platinum, is fused with bisulphate of potash in a platinum crucible to dissolve out the rhodium; an operation which is repeated until the bisulphate no longer acquires a red or pink colour. The rhodium is determined by one of the methods before described. (D.) The residue undissolved by bisulphate of potash, which is peroxide of iridium with a little platinum, is treated with aqua regia, and the peroxide reduced by hydrogen gas, in the same manner as described in a previous part of the analysis. (E.)

(K.) The concentrated solution from which the sulphurets were precipitated contains a small quantity of iridium and rhodium, protochloride of iron, and a trace of manganese. The quantity of the last is commonly too minute to be determined. The iron is peroxidized by heating with nitric acid, and weighed as peroxide, obtained by precipitation by ammonia. But this peroxide of iron contains a small quantity of a combination of iridium and rhodium, to separate which, after weighing the peroxide, it must be reduced by hydrogen gas: the reduced metal is treated with hydrochloric acid to dissolve iron, and the black undissolved portion is collected on a filter, ignited with exposure to the air, and weighed; its weight, deducted from that of the peroxide of iron previously obtained, leaves the quantity of the latter in a pure state.

The solution filtered from the precipitate by ammonia is mixed with carbonate of soda in sufficient quantity to de-

compose the ammoniacal salts, and evaporated to dryness. On treating the residue with water after a gentle ignition, peroxides of iridium and rhodium remain undissolved, but the amounts of these are in general too small for separation.

To facilitate the conception of this intricate analysis, I have annexed the following tabular view of the several steps in the process.

The weighed ore is dissolved in aqua regia, distilling the acid:

(a.) The distilled liquid contains *osmium*; dilute with water, neutralize most of the excess of acid by ammonia, and precipitate osmium, as sulphuret, by sulphuretted hydrogen; weigh the sulphuret of osmium.

(b.) Dilute the concentrated solution in the retort, and collect the undissolved grains of osmium and iridium (with sand) in a weighed filter; weigh this insoluble matter.

Mix the filtered solution with twice its bulk of alcohol of sp. gr. 0.833, add chloride of potassium, filter, and wash with alcohol.

The operation is now divided into, I. the examination of the precipitate; and, II. the examination of the alcoholic solution.

I. EXAMINATION OF THE PRECIPITATE PRODUCED BY CHLORIDE OF POTASSIUM.

The precipitate consists of the double chloride of platinum and potassium, the double chloride of iridium and potassium, and the double chloride of rhodium and potassium. Mix it with carbonate of soda, and ignite the mixture in a porcelain crucible; treat the ignited mass first with water, and afterwards with dilute hydrochloric acid. Wash, dry, and weigh the insoluble residue of metallic platinum and peroxides of rhodium and iridium, and after weighing use it with bisulphate of potash.

Platinum and peroxide of iridium remain undissolved in the bisulphate. Treat the saline mass with water, and wash, ignite, and weigh the insoluble residue. When weighed, digest the residue in aqua regia with the addition of some chloride of sodium.

Rhodium is dissolved in the bisulphate, and may be separated from the insoluble residue by treating the mass with water. Having weighed the residue, the quantity of rhodium may be determined from the loss on the weight of the mixture of platinum and peroxides of rhodium and iridium, before fusion with bisulphate of potash. The loss is *peroxide of rhodium*.

The portion undissolved is *peroxide of iridium*, which is reduced to the metallic state by hydrogen gas, and weighed.

The solution in aqua regia contains platinum and a little iridium: add to it carbonate of soda, evaporate the mixture to dryness and ignite the residue; treat the ignited mass first with water, and afterwards with aqua regia.

Peroxide of iridium remains undissolved: it should be reduced to the metallic state by hydrogen gas, and weighed.

The weight of *platinum* is known by deducting the united weights of the peroxides of iridium and rhodium from the weight of the mixture of these with metallic platinum previously ascertained.

II. EXAMINATION OF THE ALCOHOLIC SOLUTION FILTERED FROM THE PRECIPITATE PRODUCED BY CHLORIDE OF POTASSIUM.

Transmit sulphuretted hydrogen gas in excess through the solution, and allow the liquid to remain in a warm place for twelve hours.

The precipitate which is produced contains the sulphurets of iridium, rhodium, palladium, and copper: roast it in a platinum crucible with exposure to the air, and treat the roasted mass with hydrochloric acid.

Fuse the portion insoluble in hydrochloric acid with bisulphate of potash, and treat the mass with water.

Peroxide of iridium, with a trace of platinum, remains undissolved.

Rhodium is dissolved.

Mix the solution in hydrochloric acid with chloride of potassium and nitric acid; evaporate to dryness, and treat the residue with alcohol of sp. gr. 0.833.

The insoluble portion is *chloride of potassium and palladium*.

The filtered alcoholic solution contains chlorides of copper and potassium.

Evaporate off the alcohol, and precipitate *oxide of copper* by potash.

The solution filtered from the precipitated sulphurets contains iron, iridium, rhodium, and manganese: warm the solution with nitric acid to peroxidize the iron, and then add excess of ammonia.

Peroxide of iron is precipitated, together with a small quantity of a combination of iridium and rhodium.

The solution contains iridium and rhodium; add to it carbonate of soda, evaporate the mixture to dryness, ignite the residue, and treat it with water. *Peroxides of iridium and rhodium* remain undissolved, accompanied by a trace of manganese.

IX. TIN.

Tin is best weighed in the state of peroxide. If the metal is contained in solution in the state of *protoxide*, or *protochloride*, strong nitric acid should be added to the solution, and the mixture evaporated nearly to dryness at a boiling heat; the protoxide of tin is thereby converted into the insoluble peroxide. Although peroxide of tin is insoluble in nitric acid after being boiled in the liquid, it is still slightly soluble in hydrochloric acid. If the latter acid is present, therefore, it should be expelled from the liquid by boiling, with the occasional addition of some nitric acid; the hydrochloric and nitric acids suffer mutual decomposition, with the formation of nitrous acid and free chlorine, both of which are expelled by the ebullition. When the conversion of protoxide into peroxide of tin is complete, which is perceived by the cessation of nitrous vapours, the liquid may be diluted with water, and the peroxide collected on a filter. Peroxide of tin must be ignited previous to being weighed.

If tin exists in solution in the state of *peroxide*, or *perchloride*, the solution is boiled with nitric acid in the same manner; peroxide of tin is thus precipitated, which may be filtered, ignited, and weighed.

If the substance of which the tin is an element is a metallic alloy, it should be boiled in moderately strong nitric acid, when the oxides of all metals which are soluble in that acid are formed and dissolved, while the tin is converted into insoluble peroxide. The liquid is evaporated until nearly all the excess of acid is expelled, diluted with water, and filtered to collect the peroxide of tin, which is ignited and weighed.

As tin is precipitable by sulphuretted hydrogen, whether existing in the state of protoxide or peroxide, that reagent affords the means of separating tin from the alkalis, earths, and the metals not precipitated by sulphuretted hydrogen from their acid solutions. To estimate tin in such a case, the precipitated sulphuret, when washed and dried, is introduced

into a flask, and treated with fuming nitric acid, proper means being adopted to prevent loss of the solution by projection, from the intensity of the action. The tin is thus converted into peroxide, and the sulphur into sulphuric acid. The acid liquid is poured into a platinum capsule or crucible, and evaporated carefully to dryness. When all the nitric and sulphuric acids are expelled, the residue should be heated to redness, after which, pure peroxide of tin remains. In case a little sulphuric acid should be retained by the peroxide, it is recommended, after the first weighing, to place a fragment of carbonate of ammonia in the crucible, and to heat again to redness and weigh.

The *analysis of the native oxide of tin* is performed in the following manner:—Mix the finely powdered ore with five or six times its weight of caustic potash or soda, heat the mixture to dull redness in a closely covered silver crucible for half an hour, and dissolve the resulting mass, which is usually green, in hydrochloric acid. Evaporate off most of the excess of acid, dilute the remaining liquid with water, and precipitate tin as sulphuret by sulphuretted hydrogen. Treat the precipitated sulphuret of tin with fuming nitric acid in the manner before described. The liquid filtered from the sulphuret contains protochloride of iron; peroxidize the iron by boiling the solution with nitric acid, neutralize with ammonia, and precipitate peroxide of iron by a neutral alkaline benzoate or succinate. (Page 154.) From the liquid filtered from the precipitate of benzoate or succinate of iron, manganese may generally be precipitated by hydrosulphate of ammonia.

X. ANTIMONY.

In the estimation of antimony that metal is best precipitated as sulphuret, from its slightly acidified solutions, by means of sulphuretted hydrogen. It is indifferent, in this operation, whether the antimony exists in the state of oxide or the corresponding chloride, or as antimonious or antimonie acid. Before applying sulphuretted hydrogen gas, the liquid

should be diluted with water; but if the solution is neutral, or nearly so, dilution with water may cause the precipitation of a subsalt of antimony, an inconvenience that is avoided by first adding a considerable quantity of tartaric acid to the solution, after which, it may be diluted with water, without the production of any precipitate. The liquid, when saturated with sulphuretted hydrogen, should be left in a warm place exposed to the air, until the odour of the gas entirely disappears. The necessity of getting rid of the excess of sulphuretted hydrogen arises from the circumstance that water saturated with that gas possesses the property of retaining considerable traces of sulphuret of antimony in solution, which are deposited, however, when the sulphuretted hydrogen is dissipated.

The precipitated sulphuret is collected on a weighed filter, dried and weighed with the filter in the usual manner. If the precipitate which is weighed is the pure sulphuret of antimony (Sb S_3) corresponding to the oxide of antimony (Sb O_3), we can, from its weight, calculate at once the amount of metal or oxide it represents. But that sulphuret is rarely precipitated in a state of sufficient purity for the calculation, it being generally contaminated with free sulphur, and sometimes with the higher sulphurets of antimony, namely, those corresponding to antimonious and antimonie acids, when it is obviously impossible to estimate, from the weight of the sulphuret, the amount of metal it contains. To ascertain, therefore, the purity of the precipitated sulphuret, after having weighed it, digest a small portion in concentrated hydrochloric acid: if it dissolves completely, with evolution of sulphuretted hydrogen, it is the pure sulphuret corresponding to the oxide; but if it leaves sulphur undissolved, it contains either free sulphur or a higher sulphuret of antimony. In the latter case, it is necessary that another operation should be performed to ascertain the real amount of antimony contained in the sulphuret. A known weight is introduced into a flask, and fuming nitric acid is added, drop by drop, proper means being taken to prevent loss of the liquid by projection.

When a considerable quantity of nitric acid has been thus gradually added, hydrochloric acid is applied, and the mixture is digested at a gentle heat until the whole or the principal portion of the sulphur is dissolved. The liquid is diluted, and the undissolved sulphur, if any remains, is collected in a weighed filter, dried carefully, and weighed on the filter in the ordinary manner. The sulphuric acid into which the remainder of the sulphur has been converted is precipitated by chloride of barium, after an addition of tartaric acid, to prevent the precipitation of a subsalt of antimony, on dilution with water. From the weight of the precipitated sulphate of barytes, that of the sulphur it contains is calculated, which, added to that already weighed, and the sum deducted from the weight of the sulphuret of antimony employed, leaves the amount of metallic antimony in the sulphuret. Another calculation is then necessary to ascertain how much antimony was contained in the entire amount of the sulphuret precipitated by sulphuretted hydrogen, as a portion of it only was operated on.

Separation of antimony from other metals.—From those metals whose sulphurets are insoluble in hydrosulphate of ammonia, (*manganese, iron, zinc, cadmium, cobalt, lead, bismuth, silver, mercury, and copper,*) antimony may be separated by the ready solubility of its sulphuret in that liquid. The metallic alloy, or mixture of oxides, is dissolved in concentrated hydrochloric acid or aqua regia, and the solution is supersaturated, first with ammonia, and afterwards with an excess of hydrosulphate of ammonia, which should contain its full dose of sulphur. After a gentle heating, the liquid is filtered, and the precipitate is washed with water to which some hydrosulphate of ammonia has been added. The filtered liquid is a solution of sulphuret of antimony in hydrosulphate of ammonia; and the former may be precipitated by the addition of acetic acid, of which a slight excess should be used. The quantity of antimony in the precipitate is then determined by the process before described.

In an alloy, antimony may be separated from many other metals by a process founded on the volatility of its chloride.

The alloy, reduced to a state of powder, is introduced into the bulb of the tube *d*, of the apparatus figured at page 166., and chlorine gas is caused to pass through the tube. The wash-bottle *b* may contain water to arrest hydrochloric acid. The bottle *f* is one half or two thirds filled with a weak solution of tartaric acid, to which a little hydrochloric acid is added. When the apparatus is full of chlorine, heat should be applied to the bulb; chloride of antimony then distils into the receiver, affording a limpid solution, if sufficient tartaric and hydrochloric acids are present. When all the chloride of antimony is distilled, the tube is divided near the bulb, and the adhering chloride of antimony is washed into the receiver. Through the solution thus obtained, sulphuretted hydrogen gas is passed to precipitate antimony as sulphuret, and the purity of the precipitate is tested in the manner before described. The quantity of the fixed chlorides in the bulb may be estimated by first weighing the tube in its present state, then dissolving out the chlorides by water or an acid; and, lastly, weighing the empty tube when cleaned and dried. The difference in the two weighings is, of course, the weight of the fixed chlorides.

From *tin*, the sulphurets of which metal are soluble in hydrosulphate of ammonia, antimony may be separated by precipitating it in the metallic state from the solution of the two chlorides by a bar of tin with the assistance of heat. The precipitated antimony may be collected, dried, and weighed; and the united weights of the two metals being previously ascertained, the amount of tin originally present is known from the loss. If the metals exist in solution, and their united weight is not known, they may both be pre-precipitated together in the metallic state by a bar of zinc: after being collected and weighed, the mixed metals are dissolved in hydrochloric acid with a few drops of nitric acid. Metallic antimony may then be precipitated by tin, collected on a weighed filter, dried, and weighed.

Estimation of antimonious acid. — If the antimonious acid is dissolved in an alkaline liquid, the excess of alkali is

first neutralized by adding nitric acid until a precipitate begins to appear. Antimonic acid is then precipitated in the form of the antimoniate of the suboxide of mercury, by adding a solution of the protonitrate of mercury. A few drops of ammonia may be added to the mixture to insure the complete precipitation of the antimoniate. The precipitate is collected, dried, and heated to redness. By ignition, it is decomposed, oxide of mercury being given off, and pure antimonic acid remaining, in a fit state to be weighed.

XI. MOLYBDENUM.

To estimate molybdenum, or molybdic acid, the concentrated solution of the substance is treated with an excess of hydrosulphate of ammonia; the sulphuret of molybdenum is thus formed, and is dissolved by the excess of hydrosulphate. From the solution, when diluted, the tersulphuret of molybdenum is precipitated by adding hydrochloric or acetic acid, and the mixture is digested at a gentle heat, until the odour of sulphuretted hydrogen is no longer perceptible. The precipitated tersulphuret, when calcined in a small retort, parts with sulphur, being converted into the grey bisulphuret, in which state it is weighed. From the weight of the sulphuret, the amount of molybdenum it contains, or molybdic acid it represents, may be estimated.

Analysis of molybdate of lead.—Dissolve the powdered mineral in dilute hydrochloric acid, with the assistance of heat, and collect the chloride of lead which is formed in a weighed filter; after washing with water, and drying, weigh the chloride in the filter with the usual precautions. Neutralize the filtered liquid by ammonia, add an excess of hydrosulphate of ammonia, and digest the mixture for an hour in a closed flask. The lead not precipitated as chloride now falls as sulphuret, which may be collected and estimated by conversion into sulphate of lead (page 172.). To the liquid filtered from the sulphuret of lead, which is a solution of sulphuret of molybdenum in hydrosulphate of ammonia, add hydrochloric or acetic acid, and allow the mixture to

digest at a gentle heat until the odour of sulphuretted hydrogen is no longer perceived. Calcine the precipitated tersulphuret of molybdenum, when dry, in a small retort, and weigh it as the bisulphuret.

XII. TUNGSTEN.

Estimation of tungstic acid. — The ordinary process for estimating tungstic acid in a solution resembles that by which molybdic acid is estimated. An excess of hydrosulphate of ammonia is added to a concentrated neutral or alkaline solution containing the tungstic acid; sulphuret of tungsten is thus formed, and is dissolved in the excess of hydrosulphate of ammonia. The sulphuret in solution is now precipitated by dilute nitric acid, and the mixture is exposed to a gentle heat to expel the excess of sulphuretted hydrogen, on which all the tungsten is precipitated as tersulphuret, of a yellow colour. The precipitate is collected on a filter, washed with water slightly acidified by hydrochloric acid, dried, and ignited strongly in a platinum crucible with exposure to the air. The sulphuret thereby becomes oxidized and converted into tungstic acid, whose weight may be determined. When tungstic acid exists in a solution, with no other base but ammonia present, its weight may be determined by evaporating the solution to dryness, and calcining the residue, after which pure tungstic acid remains.

Tungstic acid may be precipitated from its solution, for the purpose of weighing, by means of protonitrate of mercury, in precisely the same manner as antimoniac acid. (See *Estimation of antimoniac acid*, p. 199.)

Analysis of tungstate of lime. — The analysis of tungstate of lime may be performed in the following manner: — The mineral is reduced to a fine powder, and digested at a gentle heat in three times its weight of concentrated nitric acid until nearly all the acid is evaporated. If the digestion and evaporation are repeated, the tungstate of lime is entirely converted into nitrate of lime, and the tungstic acid is set at liberty. When the excess of nitric acid is eva-

porated off, alcohol is added to the dry mass to dissolve nitrate of lime, and the residue, which is tungstic acid, is dissolved in ammonia. If any thing remains undissolved after the successive action of alcohol and ammonia, it is probably silica or some stony matter foreign to the mineral. To estimate the lime, sulphate of ammonia is added to the alcoholic solution to precipitate sulphate of lime, which is dried, ignited, and weighed. The solution of tungstic acid in ammonia, when evaporated to dryness and calcined, leaves pure tungstic acid.

Analysis of Wolfram (tungstate of iron and manganese). — The finely powdered mineral is fused in a platinum crucible with twice its weight of carbonate of soda, and the fused mass is digested in water until nothing more is dissolved by that liquid. On treating the residue insoluble in water with hydrochloric acid, oxides of iron and manganese dissolve; these oxides are separated from each other by an alkaline succinate or benzoate (p. 154.). From the alkaline solution of the fused mass in water, tungstic acid is precipitated by hydrochloric acid, washed, and redissolved in solution of ammonia. On evaporating this solution to dryness, and calcining the residue, pure tungstic acid remains, which may be weighed. Or instead of that, the tungstic acid may be precipitated by protonitrate of mercury, when the tungstate of the suboxide of mercury falls, which leaves a residue of pure tungstic acid when heated to redness in a fit state for being weighed.

XIII. ARSENIC.

Estimation of arsenic in alloys, arseniurets, arsenites, and arseniates. — To estimate arsenic, the solution in an acid of the substance to be analyzed should be diluted with water, and the arsenic precipitated from it as sulphuret by sulphuretted hydrogen. The precipitated sulphuret is that which corresponds to the degree of oxidation of arsenic in the solution: if the arsenic in solution is in the state of arsenious acid, then sulphar arsenious acid is precipitated; but if as arsenic

acid, it is sulpharsenic acid which is formed. Frequently the precipitate consists of a mixture of the two sulphurets. When the subject of analysis is an arseniuret, that is, a combination of arsenic with another metal, it should be dissolved by aqua regia: the whole of the arsenic is then converted into arsenic acid. If the compound for analysis is a soluble arsenite or arseniate, it should be dissolved in water, and the solution acidified strongly by hydrochloric acid before applying the gas. As a solution of arsenious acid is precipitated by sulphuretted hydrogen with greater facility than a solution of arsenic acid, it is advisable to reduce arsenic acid to the state of arsenious acid, before exposing it to sulphuretted hydrogen. This may be effected by means of sulphurous acid at a boiling heat. (See p. 119.)

When saturated with sulphuretted hydrogen, the liquid should be gently heated in an open vessel until the excess of the gas is dissipated, as the aqueous solution of sulphuretted hydrogen is capable of dissolving a small quantity of the sulphurets of arsenic.

If no doubts of the purity of the precipitate are entertained, that is, of its containing no free sulphur, and of its being either entirely sulpharsenious or sulpharsenic acid, and not a mixture of these sulphurets, then it may be collected in a weighed filter, dried, and weighed with the filter, and from the weight of the sulphuret, the quantity of metallic arsenic it contains, or arsenious or arsenic acid it represents, may be deduced by calculation. But it rarely happens that the sulphuret is obtained sufficiently pure for the calculation: for as the mere exposure of solution of sulphuretted hydrogen to the air causes a precipitate of sulphur, it is scarcely possible to obtain either of the sulphurets quite free from that element. Another operation must then be performed to ascertain the true amount of arsenic in the precipitate, which is precisely analogous to that already described for a similar purpose under antimony (page 197.). After the sulphuret is collected on a weighed filter, washed and dried, it is weighed together with the filter, and as much as can easily be removed is introduced into a flask, and digested with aqua regia. The

remaining sulphuret with the filter is weighed, after another drying, to know the amount introduced into the flask. By digestion with aqua regia, the arsenic becomes converted into arsenic acid, and a portion or the whole of the sulphur into sulphuric acid. After a long digestion, to insure perfect decomposition of the sulphuret, the liquid is diluted, and the undissolved sulphur (if any) is collected in a weighed filter, dried and weighed. The sulphuric acid in the solution is estimated as sulphate of barytes, the liquid being made pretty strongly acid to prevent the precipitation of arseniate of barytes. The sulphate of barytes being weighed, the quantity of sulphuret it contains is calculated, and added to that already collected: the sum, deducted from the weight of the sulphuret employed, leaves the amount of metallic arsenic.

After having precipitated arsenic in the state of sulphuret from a solution of arsenious acid, by means of sulphuretted hydrogen, and determined the weight of the precipitate, the small quantity of free sulphur which is mixed with it may often be perfectly separated by treating the precipitate with ammonia, which dissolves the sulphuret of arsenic without affecting the sulphur. The quantity of sulphur can then be readily determined. (Rose.)

In this manner the acids of arsenic may be separated from the *alkalies*, *earths*, and all metals not precipitated by sulphuretted hydrogen from their acid solutions.* From the greater number of those metals the solutions of which are precipitated by sulphuretted hydrogen, arsenic, like antimony, may be separated by the ready solubility of its sulphurets in hydrosulphate of ammonia. To the solution of the substance, add first an excess of pure ammonia, and afterwards an excess of hydrosulphate of ammonia; sulphurets of

* It has been observed by M. Wöhler, that when sulphuretted hydrogen gas is passed through a solution in a mineral acid of arsenic acid and oxide of zinc, the precipitate which is produced is a combination of sulpharsenic acid and sulphuret of zinc ($\text{Zn S} + \text{As S}_3$), whatever excess of free acid may exist in the solution. But if the arsenic acid is reduced to the state of arsenious acid before transmitting sulphuretted hydrogen (which may be effected by means of sulphurous acid, page 119.), sulpharsenious acid is precipitated in a pure state, all the zinc remaining in solution.

the various metals are thereby formed, sulphuret of arsenic is redissolved, while the other sulphurets remain insoluble. When cold, the precipitate is collected on a filter, and washed with water containing a little hydrosulphate of ammonia. On acidifying the filtered liquid with acetic acid, sulphuret of arsenic and free sulphur are precipitated, and sulphuretted hydrogen is evolved. After the liquid has been digested at a gentle heat until the odour of sulphuretted hydrogen is no longer perceptible, the precipitate may be collected on a weighed filter, and its proportion of arsenic determined by one of the processes above described.

Alloys of arsenic with metals whose chlorides are not volatile at a red heat, may be analyzed by exposing the alloy to chlorine gas, and distilling the volatile chloride of arsenic in precisely the same manner as the chloride of antimony in the analogous method of analyzing alloys of antimony.

The method of separating arsenic from *antimony* is not very exact. If the substance for analysis is an alloy consisting of no other metal than arsenic and antimony, the former may be separated from the latter by distillation in an atmosphere of hydrogen gas. The alloy is powdered and introduced into the bulb of the tube (*fig.* 19. page 160.); dry hydrogen gas is passed through the apparatus, and the heat of a spirit lamp is applied to the bulb until all the arsenic is distilled and driven out of the tube. The weight of the residue of metallic antimony deducted from the original weight of the alloy gives that of the arsenic expelled.

When arsenic and antimony occur mixed with other metals, or in a state of solution, the above process cannot be practised. In such a case, the solution of the substance (previously mixed with tartaric acid to prevent the precipitation of a subsalt of antimony) is diluted with water, and the sulphurets of both metals are precipitated by sulphuretted hydrogen. The two sulphurets should be well mixed by agitation, and then collected in a weighed filter, washed, dried and weighed. Two operations are now to be performed on the precipitate; the first to ascertain its amount of sulphur, and the second to ascertain the relative propor-

tions of antimony and arsenic present. 1. The weight of the whole precipitate being ascertained, about one half of it is removed into a flask, and the filter with the remainder is warmed and weighed, to learn the quantity introduced into the flask. That portion is very cautiously treated with aqua-regia, beginning by adding fuming nitric acid drop by drop, and afterwards adding hydrochloric acid with the same caution. Tartaric acid is added to the solution thus formed, which is diluted with water, and any sulphur which may remain undissolved is collected and weighed. The sulphuric acid in solution is then estimated as sulphate of barytes, the proportion of sulphur in which added to that previously weighed, and the sum deducted from the weight of the sulphurets employed, gives the proportion of mixed antimony and arsenic. 2. To obtain the relative proportions of the two metals, a known weight of the other portion of the precipitated sulphurets is heated in a current of hydrogen gas in the apparatus (*fig.* 19. p. 160.), when sulphur from the sulphuret of antimony is first disengaged, and afterwards sulphuret of arsenic sublimes, leaving a residue of metallic antimony, which may be weighed. Having previously obtained the united weight of both metals, the weight of the tin can now be calculated. When arsenic and antimony are mixed with metals which are precipitated by sulphuretted hydrogen from their acid solutions, but whose sulphurets are insoluble in hydrosulphate of ammonia, the two sulphurets may be obtained together, separate from the other sulphurets, in the same manner as arsenic or antimony alone is separated from such metals.

SECTION V.

NON-METALLIC BODIES.

I. SILICON.

Estimation of silicic acid (silica).—For the purpose of estimation, silicic acid may be separated from the solution in which it is contained, by first rendering the latter strongly acid, if it is not already so, and then evaporating the liquid to dryness. Hydrochloric acid is generally employed to acidify the solution of silicic acid. During evaporation, the silicic acid is deposited, most frequently in the form of an insoluble jelly, but occasionally in a pulverulent state: it is not, however, completely separated until the liquid is brought to perfect dryness, and the residue treated with a dilute acid to remove all soluble matters. Silicic acid then remains undissolved, generally in a state of purity, and may be collected on a filter, washed, dried, and ignited in a platinum crucible. As thus obtained, silicic acid is highly hygrometric; hence the crucible in which it is contained should be kept well covered while being cooled and weighed.

ANALYSIS OF NATURAL SILICATES.

The first ingredient of silicates whose weight should be determined in the analysis of these bodies is *water*, to estimate which a known weight of the silicate previously deprived of all its hygrometric moisture by gentle drying (page 128.) is heated to redness in a weighed platinum crucible for about half an hour. The loss of weight sustained by the ignition indicates the amount of water contained in the silicate. For the subsequent operations, it is better to employ another portion of the mineral, as silicates which have undergone ignition do not dissolve so readily as before being heated.

For the purpose of analysis, silicates may be conveniently divided into two classes; those decomposable by acids, and those undecomposable by acids.

I. SILICATES DECOMPOSABLE BY HYDROCHLORIC ACID.

Allophane	Gadolinite	Nosian
Analcime	Gehlenite	Okenite
Anorthite	Haüyne	Orthite
Apophyllite	Helvine	Pectolite
Botryolite	Heulandite	Potash-harmotome
Calamine	Ilvaite	Pyrosmalite
Cerine	Laumonite	Scapolite
Cerite	Lazulite	Scolezite
Chabasie	Leucite	Siliceous oxide of copper
Cronstedtite	Meerschaum	Sodalite
Datholite	Mesole	Stilbite
Dioptase	Mesolite	Titanite (Sphene)
Epistilbite	Natrolite	Wollastonite.
Eudialite	Nepheline	

II. SILICATES NOT DECOMPOSABLE BY HYDROCHLORIC ACID.

Acmite	Emerald	Petalite
Albite	Epidote	Pinite
Andalusite	Euclase	Prehnite
Autophyllite	Felspar	Pyroxene
Axinite	Garnet	Serpentine
Barytes-harmotome	Hornblende	Soapstone
Carbonated manganese	Idocrase	Soda-spodumene
Carpholite	Labradorite	Spodumene
Chlorite	Lepidolite	Staurolite
Chondrodite	Mica	Talc
Diallage	Obsidian	Topaz
Dichroite	Olivine	Tourmaline.

Except, however, in estimating the amount of alkali contained in the mineral, the course to be pursued in the analysis of a silicate of one class differs from that of a silicate of the other class only at the commencement of the process.

(1.) The following is the method commonly practised in the analysis of a silicate decomposable by hydrochloric acid. The mineral being reduced to a very fine powder, is dried at a gentle heat in a platinum crucible to expel hygrometric moisture, weighed and digested in hydrochloric acid in a porcelain basin. The bases formerly united to silicic acid thereby become chlorides, and silica, having the appearance of a jelly, is set free. If the decomposition is complete, no

gritty particles of the mineral are perceived on drawing the point of a glass rod across the bottom and sides of the porcelain basin.

The solution thus obtained is evaporated to perfect dryness, the residue moistened with hydrochloric acid, and then digested in hot water. The chlorides dissolve, leaving a residue of pure silicic acid, which may be filtered, washed, ignited, and weighed.

(2.) Silicates not decomposable by hydrochloric acid must be fused with an alkaline carbonate, a caustic alkali, carbonate of barytes, or quick-lime, and afterwards dissolved in an acid. As this operation has already been fully described when treating of the "qualitative analysis of silicates," page 79., any further notice of it is unnecessary.

After separation of the silica, the remaining steps in the analysis of silicates of both classes differ according to the nature of the constituents. Supposing the mineral to contain all the bases which ordinarily enter into the composition of silicates (page 80.), the usual course is, first to add a slight excess of ammonia to the solution filtered from the silicic acid, on which *alumina* and *peroxide of iron*, with traces of oxide of manganese and magnesia, are precipitated, methods of separating which have already been described (pages 154. and 155.). From the liquid filtered from those oxides, *lime* is precipitated by oxalate of ammonia (page 138.), and the oxalate of lime being separated, *manganese* is then thrown down as sulphuret by hydrosulphate of ammonia (page 151.); *magnesia* may be precipitated afterwards by phosphate of soda and ammonia. For details of the methods of estimating and separating these bases, I must refer to what has been previously said on the subject, under each particular base.

When magnesia and manganese occur together, a better method than the above may be adopted, as an example of which the following analysis of the mineral *colophonite* is subjoined (Liebig. *Handwörterbuch*, p. 343.).

The constituents of that mineral are:—silica, alumina, magnesia, lime, peroxide of iron, and protoxide of manganese. After fusion with an alkaline carbonate and separation of

silica in the ordinary method, the solution is mixed, first, with chloride of soda, and then with bicarbonate of potash.

The solution contains *lime* and *magnesia*.

The precipitate contains alumina, peroxide of iron, and peroxide of magnesia. Treat it with solution of potash to dissolve out alumina. Dissolve the residue, which consists of peroxide of manganese and peroxide of iron, in hydrochloric acid, and saturate the solution with carbonate of barytes in the cold.

The solution contains manganese and barytes: precipitate *manganese* by hydro-sulphate of ammonia.

The precipitate is peroxide of iron mixed with carbonate of barytes. Dissolve in hydrochloric acid, and precipitate *peroxide of iron* by ammonia.

Estimation of alkalis in silicates.—The amount of alkali contained in silicates decomposable by acids, and not containing magnesia, may be easily determined in the following manner:—The mineral is dissolved in hydrochloric acid, and silica is separated in the usual manner. To the solution, which contains the chlorides of the bases formerly united to silica, carbonate of ammonia is added, and the liquid filtered from the precipitate. In the absence of magnesia, all the earthy bases are thereby precipitated, and the filtered solution, when evaporated to dryness and the residue calcined, yields the alkali in the state of chloride, fit for weighing.

But it is obvious that when fusion with an alkali is necessary to procure solution and the ordinary method is followed, the estimation of alkali will be extremely difficult, if not altogether impracticable. Other means of effecting decomposition are then adopted in the determination of the alkali, for which another portion of the mineral must be employed, all the other constituents having been previously estimated by the usual process. Several modes are proposed of procuring the decomposition of silicates by means of substances, of which the presence would offer no impediment to the estimation of the alkali; as by means of hydrofluoric acid, by fusion with carbonate of barytes or caustic barytes, and by

oxide of lead, each of which may be removed from the solution of the ignited mass in an acid, without interfering with the estimation of the alkali.

A method which gives a very satisfactory result consists in heating to whiteness in a platinum crucible for an hour an intimate mixture of one part of the silicate with about six parts of finely powdered quick-lime. The silicic acid before in combination with alkali then unites with lime, and the alkali is therefore set free. The mass, which coheres considerably from the heating, is removed from the crucible after the ignition, and carefully powdered in a glazed mortar, placed on a sheet of paper to preserve any particles which may be projected. The powder is then transferred to a basin, and digested in water for ten or twelve hours at a moderate heat; the solution thus obtained is filtered, and the undissolved lime washed. The filtered liquid is merely a solution of the alkali in lime-water, from which the former may be obtained in the following manner:—Carbonic acid gas is passed through the liquid, until the carbonate of lime at first precipitated is re-dissolved; the liquid is boiled to re-precipitate carbonate of lime (by dissipating the excess of carbonic acid gas), and filtered. The filtered solution is then slightly acidified by hydrochloric acid, evaporated to dryness, and the alkali weighed as chloride (page 130.). Instead of performing the latter part of the operation as indicated, the calcined mass may be treated with hydrochloric acid, by which both earths and the alkali are dissolved, and the solution precipitated by an excess of carbonate of ammonia; nothing then remains in solution but the alkali which is to be estimated and ammoniacal salts. The solution is evaporated to dryness, the residue is calcined, and the alkali weighed as chloride. The large quantity of ammoniacal salts present renders it difficult to effect the calcination of the residue without some loss of the alkali by projection.

The silica obtained in the analysis of silicates should always be tested as to purity after being weighed. This is best done by fusing a small quantity in a bead of carbonate

of soda on a piece of charcoal before the blowpipe. If a colourless and transparent glass is obtained, the silica is pure, or nearly so; but if an opaque bead is produced, the silica is not free from foreign earthy matter.

II. SULPHUR.

Estimation of sulphuric acid. — Few bodies can be determined with greater accuracy than sulphuric acid. The solution of the substance in water is acidified by nitric or hydrochloric acid, and the sulphuric acid precipitated in the form of sulphate of barytes, by adding nitrate of barytes or chloride of barium to the solution. Before filtration, the mixture should be heated to cause the aggregation of the precipitate, otherwise the latter generally passes through the pores of the filter. The precipitate must be ignited before being weighed. From the weight of the precipitated sulphate of barytes, that of the sulphuric acid it contains is calculated. If the substance to be analyzed is insoluble in water, but soluble in dilute acids, pure hydrochloric or nitric acid should be used as the solvent. The sulphates insoluble, or nearly so, in water and dilute acids are, sulphates of barytes, lead, lime, and strontian.

Sulphate of lead may be analyzed by fusing it with about three times its weight of carbonate of soda in a platinum crucible, by which sulphate of soda and oxide of lead are produced. All contact with organic matter must be carefully avoided, to prevent reduction of oxide of lead to the metallic state, which would inevitably destroy the crucible, by the formation of a fusible alloy of platinum with the reduced lead. When the fused mass is treated with water, the greater portion of it dissolves, leaving a residue of oxide of lead, which may be collected on a filter, and if pure, washed, dried, ignited, and weighed. If the oxide of lead is mixed with other oxides, these are separated by processes before described. To the filtered liquid, which contains sulphate and carbonate of soda, add a slight excess of nitric acid, and estimate the sulphuric acid by precipitation as sulphate of

barytes. The aqueous solution of the fused mass generally contains a little lead, which may be precipitated as sulphuret by sulphuretted hydrogen, and weighed as the sulphate (page 172.).

Sulphate of barytes and sulphate of strontian are analyzed by mixing them in a minute state of division with three times their weight of carbonate of soda, and fusing the mixture in a platinum crucible. Sulphate of soda and carbonates of the earths are formed, the latter remaining undissolved when the fused mass is treated with water. When washed, the earthy carbonates are dissolved in dilute hydrochloric acid, and estimated in the usual manner (pages 136. and 137.). The solution filtered from the earthy carbonates is acidified by hydrochloric acid, and sulphuric acid is precipitated as sulphate of barytes by chloride of barium.

Sulphate of lime may be completely decomposed by boiling it with a solution of carbonate of potash or soda, with the formation of carbonate of lime and an alkaline sulphate. The carbonate of lime is collected on a filter, dried, and weighed after a gentle calcination, and the sulphuric acid in the filtered solution is precipitated as sulphate of barytes, the excess of alkaline carbonate being previously neutralized by hydrochloric acid. After the carbonate of lime is weighed, it should be tested for any undecomposed sulphate of lime, by dissolving it in pure dilute hydrochloric or nitric acid. If an insoluble residue remains, or if chloride of barium produces a precipitate of sulphate of barytes in the acid solution, the carbonate of lime has not been free from sulphate of lime.

From the insolubility of sulphate of barytes both in water and dilute acids, sulphuric acid in solution may be separated from almost every other substance; all the salts of barytes which are insoluble in water being soluble in hydrochloric and nitric acids, with the single exception of the sulphate.

Sulphurous acid, existing in a state of solution, may be estimated by converting it into sulphuric acid by means of strong nitric acid, by aqua-regia, or by chlorine gas, with the appli-

cation of a gentle heat; the sulphuric acid formed being afterwards weighed as sulphate of barytes.

ANALYSIS OF SULPHURETS.

The ordinary method of analyzing a metallic sulphuret is to digest the substance in nitric acid or aqua-regia; the metal thereby oxidates, and commonly dissolves, and the sulphur is converted into sulphuric acid, which may be precipitated and weighed as sulphate of barytes. In general, the metal is oxidated and dissolved long before the sulphur; and as a continuance of the digestion is tedious, from the slowness of the action, if the undissolved sulphur has a pure yellow colour, and if it does not appear to be mixed with any undecomposed sulphuret, it may be collected on a weighed filter (having first diluted the solution), washed carefully, dried, and weighed on the filter. From the filtered liquid, sulphuric acid is precipitated by chloride of barium. If the sulphuret is in a highly divided state, and the acid employed highly concentrated, the whole sulphur is sometimes immediately converted into sulphuric acid.

The metals existing in the solution filtered from the sulphate of barytes are then separated and estimated in various ways by processes already treated of. If the presence of barytes arising from the excess of chloride of barium employed interferes with the determination of these metals, the barytes may first be removed by adding sulphuric acid to the solution. Weak nitric acid should not be employed to dissolve metallic sulphurets in analysis, as an evolution of sulphuretted hydrogen gas might then be occasioned, with a consequent loss of sulphur.

In all cases in which sulphur is separated in the elementary state, especially in the analysis of metallic sulphurets, the purity of the sulphur should be tested, after being weighed, by volatilizing it in a weighed porcelain crucible. If any fixed residue remains in the analysis of a metallic sulphuret, it is commonly the oxide of the metal, and was, of course, weighed in the state of sulphuret with the sulphur.

After a strong calcination of the residue in the open air to insure perfect oxidation, and also to expel any sulphuric acid which may have been formed, it is weighed; and the nature of the oxide being known, the weight of *sulphuret* which it represents is calculated and deducted from the weight obtained of sulphur. When this oxide is dissolved in hydrochloric acid, there usually remains a trace of stony matter or silicic acid undissolved, if the substance analyzed is a natural sulphuret.

In this manner, most metallic sulphurets may be analyzed; but a small number require slight modifications of the above process. The *sulphuret of silver* must be decomposed by pure nitric acid, and not by aqua-regia: when the decomposition of the sulphuret is complete, and the liberated sulphur is separated from the solution, silver may be precipitated by hydrochloric acid; and after filtration from the chloride of silver, sulphuric acid may be precipitated by chloride of barium. *Sulphuret of bismuth* must also be treated with nitric acid, and not with aqua-regia. The liberated sulphur being collected and washed with dilute nitric acid, bismuth is precipitated from the solution by carbonate of ammonia, and sulphuric acid from the filtered liquid by chloride of barium.

Sulphuret of lead may be analyzed by digesting it in fuming nitric acid, by which it is converted into the insoluble sulphate of lead. The greater part of the excess of acid being expelled by evaporation, a few drops of sulphuric acid are added, the mixture is diluted, and the sulphate of lead collected on a filter. It should be ignited previous to being weighed (page 173.). As the *sulphurets of mercury* cannot be decomposed by nitric acid, it is necessary to use aqua-regia to dissolve these compounds. The sulphuric acid produced is precipitated by chloride of barium, and mercury is thrown down from the filtered liquid by sulphuretted hydrogen (page 184.).

The combinations of sulphur with most metals may be analyzed by heating the substance in an atmosphere of chlorine gas. In this operation the apparatus, *fig. 20.* page

166., may be employed. Chloride of sulphur and chlorides of the metals are thereby formed; the former, together with the volatile metallic chlorides, distils into the receiver, which should be kept one fourth filled with water; the fixed chlorides remain in the bulb. Before applying heat to the sulphuret in the bulb, all the air in the receiver should be displaced by chlorine. The chloride of sulphur which distils is decomposed, on coming in contact with the water in the receiver, with formation of free sulphur and hydrochloric and sulphurous acids; the latter is immediately converted into sulphuric acid by chlorine in the presence of water. The sulphur is collected in a weighed filter, and solution of chloride of barium is added to the filtered liquid to precipitate sulphuric acid. The metallic chlorides remaining in the bulb may be dissolved out by water, and estimated by the ordinary processes.

A method has been recently recommended for the analysis of metallic sulphurets, which consists in passing pure oxygen gas over the substance, heated to redness in a porcelain tube placed across a furnace: sulphurous acid is thus formed from combustion of the sulphur, and conducted into a vessel containing a strong aqueous solution of chlorine*, by which it is converted into sulphuric acid (Maugham).

The analysis of *alkaline* and *soluble earthy sulphurets* may be performed in the following manner:—A solution of chloride of copper is added to the aqueous solution of the substance to be analyzed, and the precipitate, which is sulphuret of copper, is rapidly collected on a filter, and oxidized by fuming nitric acid. When the sulphur is completely dissolved, a solution of nitrate of barytes is added to precipitate the sulphuric acid into which the sulphur has been converted, and the amount of sulphur is calculated from that of the sulphate of barytes obtained. If the alkaline sulphuret is treated at once with nitric acid, some sulphur would escape oxidation and be lost, being evolved in the state of sulphuretted hydrogen gas; hence the necessity of having the

* A solution of chloride of soda would probably be found more convenient than chlorine water in this operation.

sulphur in the state of sulphuret of copper, which does not yield sulphuretted hydrogen with strong nitric acid. The liquid, filtered from the precipitated sulphuret of copper may be treated with sulphuretted hydrogen, to separate copper; and the alkali or earth remaining in solution may be determined in the usual manner.

Instead of mixing the solution of chloride of copper with the solution of the alkaline or earthy sulphuret, sulphuretted hydrogen gas may be produced by decomposing the sulphuret by hydrochloric acid, and the gas transmitted through solutions of the copper salt, contained in a series of Woulf's bottles, the last solution being supersaturated with ammonia. When the decomposition is complete, a saturated solution of carbonate of ammonia should be introduced into the generating bottle to cause a stream of carbonic acid gas to traverse the whole apparatus and thus carry forward the remaining sulphuretted hydrogen. The precipitated sulphuret of copper is treated by nitric acid as above.

III. CHLORINE.

Analysis of chlorides. — Hydrochloric acid and chlorine may be determined with great accuracy. The solution of the substance to be analyzed is acidified by nitric acid, warmed, and the chlorine is precipitated in the form of chloride of silver by adding a solution of nitrate of silver to the liquid. The precautions to be observed in the precipitation and ignition of chloride of silver have already been mentioned under the subject of silver (page 179.). From the liquid filtered from the chloride, the excess of silver may be removed by adding hydrochloric acid, and the bases present may then be determined by the ordinary processes. If the substance is insoluble in water, but soluble in acids, such as many subchlorides, nitric acid should be employed as the solvent.

Chloride of silver and chloride of lead may be analyzed by reducing these compounds to the metallic state by hydrogen gas, in the apparatus *fig.* 19. (page 160.) with the assistance

of heat; hydrochloric acid gas is then disengaged. The degree of heat required for the decomposition of metallic *chlorides* by hydrogen gas is somewhat higher than that necessary to decompose the *oxides* of the same metals by hydrogen. When the decomposition of the chloride is complete, which is known by the absence of white fumes on holding a rod moistened with ammonia at the extremity of the apparatus, the tube *c* containing the reduced metals may be disconnected from the apparatus and weighed. The weight of the tube and substance having been previously determined, the loss of weight on reduction represents the amount of chlorine contained in the chloride.

Subchloride of mercury (calomel) may be analyzed by digestion in a solution of caustic potash; the black suboxide of mercury and chloride of potassium are then formed. The filtered alkaline solution is supersaturated with nitric acid, and the chlorine contained in it is precipitated by nitrate of silver. It is advantageous to take another portion of calomel to determine the mercury by means of protochloride of tin.

CHLORIMETRY.

Chlorimetry, or the process of the valuation of chloride of lime (bleaching powder), is an important subject, in connection with the chemical arts. Of many methods which have been proposed, that which appears entitled to preference is one in which the chlorine is determined by finding what quantity of the chloride of lime to be analyzed is required to convert a known weight of the protoxide of iron into peroxide. For this change, protoxide of iron requires half an equivalent of oxygen, which is supplied in the present process by the decomposition of half an equivalent of water by chlorine of the chloride of lime. Half an equivalent of chlorine, therefore, or 221.8 parts, effects this change upon a whole equivalent, or 439 parts of protoxide of iron, which quantity of protoxide is contained in one equivalent, or in 1728 parts of crystallized protosulphate of iron. Therefore 221.8 parts of chlorine can peroxidize 1728

parts of crystallized protosulphate of iron, or 10 grains of chlorine can peroxidize 78.1 grains of the crystallized protosulphate. Now the process consists in discovering how much of the specimen of chloride of lime is requisite to peroxidize 78.1 grains of the protosulphate, which quantity of the chloride must contain 10 grains of chlorine.

In the first place, 78 grains of the pure protosulphate (clean crystals of the salt dried by strong pressure between folds of cloth) are dissolved in about two ounces of water, and the solution is acidulated by a few drops of hydrochloric or sulphuric acid. Fifty grains of the chloride of lime are weighed out, mixed well in a mortar with about two ounces of tepid water, and the mixture poured into an alkalimeter (page 132.). The measure is filled up to 0 with the washings of the mortar, and the liquids are well mixed by agitation, the mouth of the alkalimeter being closed by the palm of the hand. This solution of chloride of lime is then gradually added to the solution of 78 grains of protosulphate of iron until the latter is completely peroxidized, which is discovered by means of the red prussiate of potash. This salt gives a precipitate of Prussian blue with a salt of the protoxide of iron only, and not with a salt of the peroxide; so long, then, as it produces a blue precipitate, protoxide of iron still exists in solution, and more chloride of lime must be added. In applying red prussiate as the test, it is convenient to have a plate spotted over with small drops of its solution, which are touched with the drop of the liquid to be tested, taken out on the point of a glass stirrer. Stopping short exactly at that point at which the red prussiate of potash ceases to afford Prussian blue, the number of measures poured from the alkalimeter is carefully observed; the richer in chlorine the specimen of chloride of lime is, the less of course is required. As this amount, whatever it may be, but which, for example, we will suppose to be 70 measures, contains always 10 grains of chlorine, it becomes a simple matter to calculate the per centage of chlorine in the specimen. For as 100 of the alkalimeter divisions contain 50 grains of the chloride, each single measure must

contain half a grain; the 70 measures poured out, contained therefore 35 grains of chloride of lime. Then, if 35 grains of the chloride contain 10 grains of chlorine, by simple proportion 100 grains of the chloride must contain 28·57 grains of chlorine. The only calculation to obtain the per centage is, to divide 1000 by one half the number of measures poured from the alkalimeter: —

$$\frac{1000}{35} = 28\cdot57.$$

Or, what is the same thing, to divide 2000 by the actual number of measures poured out.*

IV. IODINE.

Analysis of iodides. — The amount of iodine contained in iodides which are soluble in water may be estimated by precipitating the iodine in the form of iodide of silver by the nitrate of silver. The precipitated iodide is collected and ignited in the same manner and with the same precautions as the chloride of silver. It is, however, more usual to determine the amount of iodine as *loss*, that is, as the deficiency on the weight of the original substance, having ascertained the quantity of all the other ingredients of which the substance is composed. To obtain, then, the amount of base combined with iodine, the iodide may be decomposed by sulphuric acid in a porcelain capsule with the assistance of heat; hydriodic acid, sulphurous acid and iodine are evolved, and a residue remains, consisting of the sulphate of the oxide of the metal formerly united to iodine. The composition of the iodide being known, the weight of metal in the portion weighed is calculated and deducted from the original weight of the substance, to obtain the amount of iodine. In the case of iodides, whose metallic bases are precipitated from solution in an acid by sulphuretted hydrogen or hydrosulphate of ammonia, those reagents may be used to separate the metals.

* In the preceding process, Mr. W. Crum uses a stoppered phial to contain the solution of sulphate of iron, which is shaken, instead of an open jar or tumbler, and thus prevents any escape of chlorine. He can then add the chloride of lime in a dry state, from a weighed quantity, in the absence of an alkalimeter. (*Trans. Glasgow Phil. Society*).

If the iodide for analysis is insoluble both in water and acids, it should be ignited with carbonate of soda, and the heated mass afterwards digested in water. Iodide of sodium and the excess of carbonate of soda dissolve, and leave the metallic oxide as a residue, if it is insoluble in a solution of the alkaline carbonate. The oxide may be weighed, the corresponding amount of metal calculated, and deducted from the original weight of the iodide to obtain the amount of iodine.

Separation of iodine from chlorine.—As iodides rarely exist in nature unmixed with chlorides, it is important to have the means of completely separating chlorine from iodine. Many processes have been proposed for this purpose, one of the latest and best of which is the following, by M. Rose:—Both chlorine and iodine are precipitated from the mixed salts by nitrate of silver, and the mixed iodide and chloride of silver is collected, ignited, and weighed. The mixed iodide and chloride is then introduced into a tube of hard glass, having a bulb blown on its middle (such as the tube *c* of the apparatus (*fig.* 19. page 160.), and after being weighed with the tube, chlorine gas is passed over the substance. By the action of chlorine, the iodide of silver is decomposed, chloride of silver being formed, and iodine liberated, which may be driven out of the tube by the application of heat. On again weighing the tube, a loss will be experienced, which, multiplied by 1.389, gives the quantity of iodine contained in the mixed chloride and iodide of silver operated on. The number 1.389 bears the same proportion to 1 which the equivalent of iodine bears to the difference between the equivalents of chlorine and iodine. For every equivalent of iodine (1579.5) expelled by chlorine one equivalent of chlorine (442.65) is absorbed; the loss actually sustained in the experiment, therefore, is the difference between the equivalents of chlorine and iodine, or 1136.85 for every 1579.5 parts of iodine, which numbers are as 1 to 1.389. Having thus obtained the iodine, the amount of chlorine may be calculated by deducting the weight of the iodide of silver (found by calculation) from the weight of the mixed chloride and iodide of silver; the

remaining chloride of silver contains 24.67 per cent. of chlorine.

The proportion of iodine existing in iodine ley may be conveniently estimated by means of a solution composed of 1 part of crystallized sulphate of copper, and $2\frac{1}{2}$ parts of crystallized protosulphate of iron. On adding such a solution, all the iodine is thrown down from the ley as subiodide of copper, the chloride of copper formed at the same time from the chlorides present in the ley remaining in solution. The precipitate must be collected on a weighed filter, washed, dried, and weighed. It contains 66.6 per cent. of iodine.

V. BROMINE.

The amount of bromine contained in bromides may be estimated in the same manner as iodine is estimated in iodides; that is, either by precipitation, as bromide of silver, which is collected and fused with the same precautions as the chloride of silver, or by determining the amounts of all the other ingredients, and estimating bromine as the loss on the original weight of the substance. The amount of metallic base may be ascertained either by conversion into sulphate by heating the bromide with sulphuric acid in a porcelain capsule, or by separating the metal from the solution of the substance by means of sulphuretted hydrogen or hydrosulphate of ammonia. Bromides insoluble in acids may be analyzed by fusion with carbonate of soda, in a manner precisely similar to that described for insoluble iodides.

Separation of bromine from iodine.—In a mixed bromide and iodide the respective proportions of bromine and iodine may be ascertained by a method similar to that used for the analysis of mixed chlorides and iodides. The mixture of iodide and bromide of silver is obtained, its weight determined, and the mixture is heated in an atmosphere of bromine. A loss is found to occur after heating, which, multiplied by 2.627, gives the proportion of iodine.

VI. FLUORINE.

Analysis of fluorides.—The ordinary method of determining the amount of fluorine contained in fluorides is, to separate the fluorine in the state of hydrofluoric acid, and to calculate its quantity from the loss. To perform this, the dry weighed fluoride is moistened with oil of vitriol in a platinum capsule, and heated so long as acid vapours are given off. The residue, which consists of the sulphate of the oxide of the metal formerly in combination with fluorine, is weighed, and the proportion of *metal* it contains calculated; the weight of the metal deducted from the original weight of the substance leaves the amount of fluorine. If more than one base is combined with fluorine in the substance for analysis, it is necessary to undertake a full determination of the bases, and to calculate the amount of each *metal*. To obtain the amount of fluorine, the united weight of the metals is deducted from the weight of the fluorides operated on.

When the substance to be analyzed contains water, that, it is obvious, will be included in the loss, and must, therefore, be estimated by another operation before the fluorine can be determined. The amount of water contained in many fluorides may be estimated by heating the substance alone to redness in a platinum crucible. The whole of the water may then be given off in a pure state, and its amount is learned by the loss in weight undergone by heating. But it frequently happens, that besides the water a little hydrofluoric acid is disengaged, which vitiates the determination of the former. In such a case, the weighed fluoride, in fine powder, is intimately mixed with freshly calcined and powdered protoxide of lead, the mixture is introduced into a small hard glass retort, and covered with a little more pure oxide of lead. The retort, with its contents, is weighed, and heated to redness to expel water, which is given off without a trace of acid, the latter being retained by the oxide of lead. The vapour of water in the retort at the close of the operation is

removed by carefully sucking it out with the mouth by means of a narrow tube introduced into the retort through the neck. The loss in the weight of the retort indicates the amount of water expelled. By deducting the water from the united loss of fluorine and water previously arrived at by the determination of the bases, the amount of fluorine is ascertained.

VII. PHOSPHORUS.

Estimation of phosphoric acid.—In the analysis of combinations of phosphoric acid with substances, the weights of which are easily determined, it is usual, having obtained the amount of every other ingredient, to consider the deficiency on the weight of the matter employed as representing the phosphoric acid.

To estimate phosphoric acid, directly, it may be precipitated from its *neutral* solutions either as phosphate of lead or as phosphate of barytes; but as this acid forms compounds of different composition with the same base, the precipitate must, in general, be subjected to analysis after its weight is determined, to ascertain the actual amount of phosphoric acid it contains. The phosphate is analyzed by ascertaining directly the proportion of base it contains, and considering the loss as the phosphoric acid.

The two phosphates mentioned are analyzed by the following processes:—*Phosphate of lead* is dissolved in dilute nitric acid, and oxide of lead is precipitated from the solution as sulphate, by sulphuric acid; some alcohol is added to the mixture to prevent the solution of any trace of the sulphate, the latter being wholly insoluble in dilute alcohol. The precipitated sulphate of lead is filtered, washed with weak alcohol, ignited, and weighed. From the quantity of sulphate, that of oxide of lead is calculated, and the phosphoric acid estimated as loss. *Phosphate of barytes* is analyzed by dissolving it in hydrochloric acid, and precipitating barytes from the solution by sulphuric acid. From the weight of the precipitated sulphate of barytes, that of the barytes is cal-

culated, which, again, deducted from the original weight of the phosphate, gives the amount required of phosphoric acid.

If phosphoric acid exists alone in solution, its amount may be determined directly, by adding, of pure and freshly calcined protoxide of lead, a known weight, which is in excess, to the solution, and evaporating to dryness. The residue, which is a mixture of phosphate of lead with the excess employed of oxide of lead, is calcined in a platinum crucible, and weighed. The increase on the quantity of protoxide of lead employed is the amount of phosphoric acid formerly contained in solution.

Phosphoric acid may be separated from many metallic oxides by fusion with an excess of carbonate of soda. The mass, after being heated, contains the metallic oxide, phosphate of soda, and the excess of carbonate of soda. If the oxide is quite insoluble in a solution of the alkaline carbonate, nothing but phosphate of soda and carbonate of soda dissolves when the dry mass is treated with water. The aqueous solution is exactly neutralized by nitric acid, and acetate of lead or chloride of barium is added to precipitate phosphoric acid. The precipitated phosphate of lead or phosphate of barytes is analyzed by means of its conversion into sulphate, as before described.

Separation of phosphoric acid from alumina. — Phosphoric acid cannot be separated from alumina by the above method, as phosphate of alumina is soluble in solution of carbonate of soda. To analyze phosphate of alumina, Fuchs recommends that a known weight of the combination should be dissolved in a solution of caustic potash, and the alumina be precipitated by a solution of silicate of potash, in the form of silicate of alumina and potash. The bulky and mucilaginous precipitate which falls is collected on a filter, washed, and digested in hydrochloric acid; the acid mixture is evaporated to dryness to separate silica. When the dry mass which remains after the evaporation is moistened with hydrochloric acid, and then treated with water, it affords an insoluble residue of silicic acid, while alumina dissolves, and may be precipitated from the solution by carbonate of ammonia.

The alkaline liquid filtered from the precipitate of silicate of alumina and potash contains all the phosphoric acid.

Separation of phosphoric acid from sulphuric acid. — If a substance is to be analyzed which contains both phosphoric and sulphuric acids, the latter may be easily separated from the former by the solubility of phosphate of barytes in acids. Either hydrochloric or nitric acid being first mixed with the solution, on adding an excess of chloride of barium, sulphate of barytes only is precipitated, which may be weighed. If the solution filtered from the precipitate of sulphate of barytes is carefully neutralized with ammonia, phosphate of barytes is precipitated, the phosphoric acid contained in which may be determined by weighing and analyzing the precipitate, in the manner before described.

M. Schulze has recently proposed a method of determining the amount of phosphoric acid contained in soils, rocks, wood-ashes, &c., founded on the insolubility of phosphate of the peroxide of iron, and phosphate of alumina in acetic acid. In soils, the phosphoric acid generally exists in the state of phosphate of lime and phosphate of magnesia. If to a solution of these phosphates in acetic acid a sufficient quantity of a solution of acetate of peroxide of iron be added, all the phosphoric acid may be precipitated, in the form of phosphate of the peroxide of iron, which may be collected and weighed. Instead of acetate of iron, acetate of alumina may be employed with a similar result. As soils usually contain more peroxide of iron and alumina than is sufficient to combine with the whole of the phosphoric acid they contain, all that is required to be done, in estimating the amount of this acid contained in a soil, is to add excess of ammonia to the solution of the earthy matter in hydrochloric acid, and to treat the precipitate with acetic acid. Nothing but phosphate of the peroxide of iron and phosphate of alumina remains undissolved, and after being weighed, these phosphates may be separated from each other by dissolving the latter in solution of caustic potash.*

* The insolubility of the phosphate of the peroxide of iron in acetic acid also affords a means of separating the peroxide from the protoxide of iron; the phosphate of the latter base being soluble in acetic acid.

VIII. BORON.

Estimation of boracic acid. — Like phosphoric acid, boracic acid is best determined by the loss experienced on the original weight of the substance containing it, when the weight of every other constituent is ascertained. The estimation of boracic acid comes thus to consist in the perfect separation and determination of the weights of all the substances with which it is associated. The separation of the metals proper from boracic acid may be easily effected by sulphuretted hydrogen or hydrosulphate of ammonia, but the separation of the earths requires particular processes. When the compounds which contain this acid are decomposable by concentrated sulphuric acid, they may be analyzed by the following method, proposed by Arfvedson. A known weight of the borate to be analyzed is mixed in a platinum crucible with four times its weight of pure fluor spar, both substances being in a state of fine powder, and sufficient oil of vitriol to make a thick paste. The mixture is heated gradually to redness, and maintained at that temperature so long as any acid vapours are given off. All the boracic acid is thus converted into the gaseous fluoride of boron, which is entirely disengaged, together with the excess of sulphuric acid. The residue consists of sulphate of lime produced from the decomposition of fluor spar, and sulphates of the bases with which boracic acid was previously in combination. This residue is then subjected to a full analysis, by processes described elsewhere; and the nature and proportions of the bases being ascertained, their united weights are deducted from the original weight of the borate operated on to obtain the amount of boracic acid.

In the manner just described may be analyzed all borates which are decomposable by sulphuric acid, and which do not contain water. The same process may be followed if the borate contains lime; the quantity of fluor spar must then be accurately weighed, and the lime contained in the substance analyzed be deduced from the excess of sulphate of lime formed over the calculated quantity produced from the quantity of fluor spar employed. (Rose.)

Instead of fluor spar, hydrofluoric acid may be used to decompose borates. The borate, in a state of fine division, is mixed with hydrofluoric and sulphuric acids in a weighed platinum crucible: the mixture evaporated to dryness and calcined to expel all the excess of sulphuric acid, leaves a residue, consisting of sulphates of the bases formerly united with boracic acid.

Borate of barytes may be analyzed by dissolving it in hydrochloric or nitric acid, and precipitating barytes from the solution, as sulphate, by sulphuric acid. The weight of the barytes contained in the sulphate being calculated, the boracic acid may be estimated as loss.

C. G. Gmelin has proposed the following process for the direct determination of boracic acid. The powdered substance to be analyzed is heated to redness with carbonate of soda, and the resulting mass is digested in water, to dissolve the borate formed during the fusion. The small quantities of silica and alumina which the water may also dissolve are precipitated by digestion with carbonate of ammonia. The filtered liquid is evaporated to dryness, and the residue treated with sulphuric acid, to decompose the borate of soda; the boracic acid thus liberated is dissolved out by alcohol. The alcoholic solution, when saturated with ammonia, evaporated to dryness and the residue calcined, leaves pure boracic acid, whose weight may be determined.

IX. NITROGEN.

Estimation of nitric acid. — If nitric acid exists in solution in a free state, and unmixed with any other acid, its amount may be determined by adding a sufficient quantity of barytes water to render the liquid feebly alkaline, and evaporating the solution of nitrate of barytes thus obtained to dryness. The excess of barytes combines with carbonic acid from the atmosphere during evaporation, to form the insoluble carbonate of barytes, which therefore remains undissolved when the dry residue is treated with water. The barytes in solution is then precipitated by sulphuric acid, and weighed as sulphate. The weight of the sulphate of barytes is, of

course, proportional to that of the nitric acid, in the ratio of single equivalents of each.

When the nitric acid exists in a state of combination, it is generally estimated as loss, the amount of the base in combination with the acid being ascertained, either by conversion into sulphate, or by other means proper for the particular base.

If the base is one which may be precipitated from its acid solutions by sulphuretted hydrogen, that re-agent is advantageously employed to separate it from nitric acid. The precipitated sulphuret being separated from the solution, the nitric acid set at liberty may be determined by adding a slight excess of barytes-water to the solution, and evaporating to dryness. The small quantity of sulphuret of barium formed from the sulphuretted hydrogen in solution is converted, during evaporation, into hyposulphite and sulphate of barytes, both insoluble salts. When the dry mass is treated with water, nothing but nitrate of barytes dissolves, which may then be converted into sulphate and weighed. When the analysis is performed in this manner, it is necessary to cease passing sulphuretted hydrogen so soon as the nitrate is entirely decomposed, as an excess of the reagent would act on the liberated nitric acid, occasioning its decomposition and consequent loss.

Such *subnitrates* as are insoluble in water may be decomposed as well as if soluble, by sulphuretted hydrogen; they should be reduced to a state of minute division, and diffused through an ounce and a half or two ounces of water. These bodies may likewise be decomposed by digestion in a solution of sulphuret of barium; by which an insoluble metallic sulphuret is produced, on the one hand, and the soluble nitrate of barytes on the other. The excess of sulphuret of barium is removed from the solution by a current of carbonic acid gas, which precipitates carbonate of barytes, and causes an evolution of sulphuretted hydrogen gas. The solution is filtered and evaporated to dryness, the residue redissolved in water, and barytes precipitated from the solution by sulphuric acid. From the amount of sulphate of barytes, that of the nitric acid may be calculated.

EUDIOMETRY.

To separate the *oxygen* and *nitrogen* gases, and to determine the amounts of *watery vapour* and *carbonic acid* contained in atmospheric air, several processes have been recommended, possessing various claims to accuracy as well as simplicity. Of those for estimating the oxygen and nitrogen the principal are the following:—

(1.) Into a measure of air observed at a known temperature and pressure, and contained in a narrow graduated tube standing over water, a stick of phosphorus is introduced and allowed to remain for twenty-four hours, or so long as any absorption of gas is seen to occur. When the white vapours have entirely disappeared, the phosphorus is withdrawn, and the amount of contraction noticed, together with the temperature and pressure, for which, if they do not agree with the observations at the time of the first measurement, corrections must be made*: the gas remaining is nitrogen. This is the simplest method, but probably the least exact.

(2.) Gay-Lussac recommends a method in which oxygen is separated from nitrogen by means of slips of copper moistened with hydrochloric acid, which absorb oxygen with rapidity.

(3.) Another simple method is that in which the oxygen is estimated by observing the contraction which occurs on the ignition of a mixture of a known measure of air with one half its bulk of pure hydrogen. The ignition may be effected either by the electric spark or by means of a pellet of clay and spongy platinum. In the latter case, combination of the hydrogen and oxygen takes place without explosion; hence the experiment may be performed in a common graduated tube standing over mercury. One third of the contraction represents the volume of oxygen contained in the measure of air employed. In igniting by the electric spark, Dr. Ure's eudiometer is a convenient instrument to be used. It is formed of a straight tube (*fig.* 21.), about twenty inches in length, sealed at one end, *a*, and doubly bent at the middle.

* The means of making these corrections are given in an Appendix.

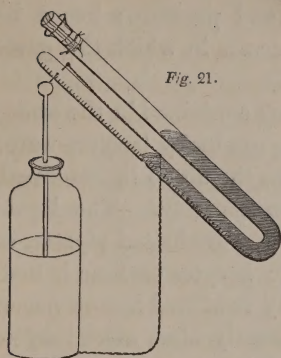


Fig. 21.

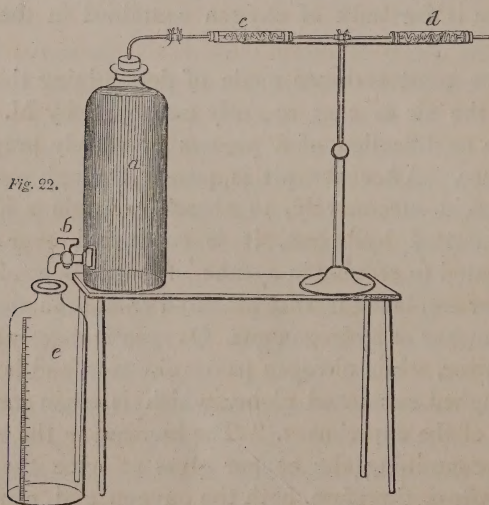
The limb with the sealed end is graduated. Two platinum wires are sealed into the glass near the closed end, with their extremities within the tube about one tenth of an inch apart. On placing one of these wires in communication with the knob of a charged Leyden jar, and the other with the ground, the spark passes within the tube. Having prepared a mixture of air and hydrogen in known proportions, enough of this mixture is transferred into the tube at the water or mercurial trough, to occupy about three inches of the sealed limb; the amount of the mixture introduced is accurately observed, the liquid standing in both limbs at the same level. The mouth of the open limb is now closed by a cork which is secured by a wire, and the electric spark is taken through it. After the explosion, a contraction is found to occur, to observe the amount of which, the liquid must again be brought to the same height in both limbs. One third of the contraction is the bulk of oxygen contained in the original mixture.

(4.) The most accurate mode of determining the composition of the air is that recently employed by M. Dumas, which is a modification of a process previously proposed by M. Brunner. After the air is passed through potash and sulphuric acid successively, to absorb carbonic acid, water, and carburetted hydrogen, it is conducted over metallic copper heated to redness in a tube of hard Bohemian glass. The copper employed is that procured from reduction of the oxide by means of hydrogen gas. Oxygen is eagerly absorbed by the copper, while nitrogen passes onwards, and is received into a weighed exhausted globe, which is again weighed at the close of the experiment. The increase in the weight of the tube containing the copper gives at once the oxygen. In this process, therefore, both the oxygen and nitrogen are determined by direct weighing, and no corrections are re-

quired for changes of temperature and pressure: hence its superiority to all eudiometrical processes in which the gases are estimated by measurement.

The proportion of *carbonic acid gas* contained in the atmosphere may be determined by adding gradually barytes-water or lime-water of a known strength to the air to be examined, contained in a large globe of known capacity. The liquid is dropped into the globe by means of a graduated pipette, so long as the alkaline re-action of the barytes or lime is neutralised, which is best ascertained by reddened litmus paper. When the paper is rendered permanently blue, more barytes or lime has been added than is necessary to form carbonate with all the carbonic acid of the air. The carbonic acid is proportional to the quantity of lime or barytes neutralized.

The most ready method of determining the amount of *watery vapour* in the air is by the hygrometer; but the subject of hygrometry belongs rather to physics than to chemistry. The same object may be attained by causing a known quantity of air to pass through a tube which contains some highly hygroscopic body; a method followed by Brunner. A cylindrical bottle, *a*, (*fig. 22.*), furnished with a stop-cock *b*, near

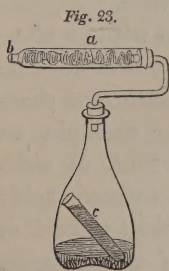


its bottom, is connected (air-tight) at top with a small tube

c, containing fragments of dry chloride of calcium; and that is connected, by means of a caoutchouc joint, with the tube *d*, which contains asbestos moistened with concentrated sulphuric acid, an extremely useful hygrometric agent. The tube *d* being weighed, and the apparatus mounted as in the figure, the stopcock is opened, and the water which flows out is received in the graduated vessel *e*. That vessel serves as the measure of the air which enters the bottle *a*. If passed with a moderate degree of rapidity, the air, in entering, is entirely deprived of its water by the sulphuric acid in the tube *d*; the increase in weight of which at the end of the experiment is the amount of water contained in a volume of air equal to the volume of water collected in the receiver *e*.

X. CARBON.

Analysis of carbonates.—The quantity of carbonic acid contained in all carbonates which are soluble in hydrochloric or sulphuric acid in the cold may be determined by the following simple method:—A known weight of the powdered substance is introduced into a flask similar to that represented in *fig. 23*. The flask should be thin at the bottom to allow of



the application of heat, and have the capacity of three or four ounces of water. It is fitted with a cork, which has a perforation to admit a small bent tube, and the latter is connected by means of another cork with a somewhat larger tube, *a*, containing fragments of dry chloride of calcium. The extremity *b* of this tube is drawn out so as to be capillary. The small tube *c* within the flask, sealed at one end, is intended to

hold hydrochloric or sulphuric acid to decompose the carbonate, and is of such length that it will not fall flat on the bottom of the flask, but rests against the side at an angle of about 45° with the bottom, so that on inclining the flask, all the acid contained in this tube can be made to flow out. The apparatus being arranged, the weighed carbonate is

introduced into the empty flask, with about half an ounce of water; the small tube *c*, containing sufficient hydrochloric or sulphuric acid to decompose the carbonate, is then introduced, taking care that no acid comes in contact with the carbonate, and the flask is closed by the cork attached to the chloride of calcium tube *a*. The whole apparatus is now weighed, after which the flask is inclined, in order that a little of the acid in the tube *c* may flow out and come in contact with the carbonate, which is repeated until the latter is completely decomposed. As the evolved carbonic acid gas is dried in passing through the chloride of calcium tube *a*, nothing else than this gas escapes, and the loss in weight of the apparatus at the close of the experiment is the weight of the carbonic acid required. But as the flask is then full of carbonic acid gas, which is considerably heavier than air, it should not be weighed in its present state. To get rid of the remaining carbonic acid, the flask is very gently heated, so as to fill it with aqueous vapour, and thus drive out the gas, the steam itself not proceeding further than the chloride of calcium tube. On the condensation of the steam, air enters the flask, which, when cold, is in the same condition as it was when weighed before the decomposition of the carbonate, excepting only in the loss of carbonic acid.

By such an operation as this, in conjunction with an alkalimetric experiment, (page 132.) the proportions of alkaline carbonate and caustic alkali contained in a mixture of these substances may be determined. Suppose, for example, an alkalimetric experiment to indicate 41 per cent. of soda, and the process just described 20 per cent. of carbonic acid; then, twenty parts of carbonic acid are equivalent to 28.2 of soda, and that deducted from 41 gives 12.8 of caustic soda, and consequently 48.2 per cent. of carbonate.

In the analysis of alkaline carbonates by this method, it is better to employ dilute sulphuric than hydrochloric acid, as a little hydrochloric acid gas might easily be carried away by the carbonic acid, or evaporated on heating the flask at the close of the operation; but for earthy carbonates whose sulphates are insoluble, the employment of sulphuric acid is

not admissible. Lime-stones and marls are analyzed very conveniently by this process.

Carbonates of oxides of the metals proper, which do not contain water, or any other volatile constituent, excepting carbonic acid, may be analyzed by simply heating a known weight of the substance to redness in a weighed platinum crucible. The loss in weight experienced by heating is the amount of carbonic acid.

ANALYSIS OF GUNPOWDER.

Separation of carbon from sulphur and nitrate of potash. —

The process of analyzing gunpowder consists, first, in the separation of nitrate of potash from carbon and sulphur, and, second, in the separation of carbon from sulphur. The gunpowder, when reduced to a fine powder and weighed, is boiled in about seven times its weight of water, to dissolve the nitre. The residue, which consists of carbon and sulphur, is collected on a weighed filter, washed with tepid water so long as any nitrate of potash is dissolved out; dried and weighed. The filtered solution of nitrate of potash is evaporated to dryness in a weighed platinum capsule in a water-bath, and the dry saline residue weighed. This residue generally contains a small quantity of chloride of sodium, to determine the quantity of which, the dry salt, after being weighed, is dissolved in water, and the amount of chlorine it contains is estimated by precipitating and weighing as the chloride of silver. The amount of chloride of sodium which corresponds to the weight of the chloride of silver is calculated, and deducted from the weight of the nitrate of potash previously obtained.

Various methods are practised to ascertain the relative proportions of carbon and sulphur. One consists in converting all the sulphur into sulphuric acid, which is weighed as sulphate of barytes. For this purpose, 1 part of the dry mixture of carbon and sulphur is mixed, first with 4 parts of carbonate of potash (free from sulphate), and afterwards with 8 parts of nitre, and 6 parts of chloride of sodium. When

that mixture is heated strongly in a platinum crucible, the sulphur becomes sulphuric acid, which unites with potash. After being heated, the mass is dissolved in water, and the excess of alkaline carbonate is neutralized with hydrochloric acid: sulphuric acid is then precipitated in the form of sulphate of barytes, which is washed, dried, and weighed. The quantity of sulphur which corresponds to the weight of the sulphate of barytes is calculated, and the carbon is estimated as loss.

Another method of separating the sulphur and carbon, advantageous from its allowing the weights of both these elements to be taken directly, consists in dissolving out the sulphur from the mixture by means of sulphite of potash; hyposulphite of potash is thus formed, and a residue of pure carbon remains, which is collected on a weighed filter, dried, and weighed. From the solution of hyposulphite of potash all the sulphur may be precipitated by the addition of hydrochloric acid: the precipitate is collected on a weighed filter, washed, dried, and weighed. (Pelouze.)

Two other methods are also practised; in the first of these carbon and sulphur are separated from each other by subliming the latter in an atmosphere of hydrogen, and weighing the residue of carbon; in the second, by heating the mixture in an atmosphere of chlorine, when the volatile chloride of sulphur is formed. But neither of these methods possesses any advantage over those described, while their execution is more difficult.

Separation of carbon from iron: analysis of cast-iron. — The estimation of carbon contained in cast-iron may be effected by an operation similar to that of an organic analysis. (Regnault.) From 70 to 80 grains of the specimen is reduced to powder (either by pulverizing in a mortar or by filing), and mixed with from 12 to 14 times its weight of chromate of lead, previously fused and reduced to powder. About one third or one fourth of the mixture is set aside; the remainder is mixed with as much powdered chlorate of

potash as the weight of cast-iron operated on, and this mixture is introduced into a tube of hard glass similar to that used in organic analysis, but much shorter. The remaining mixture of cast-iron and chromate of lead is now added, being placed above the mixture previously introduced, and the tube is connected with the potash apparatus employed in organic analysis. Heat is first applied to that part of the tube, the mixture in which contains no chlorate of potash, and is advanced gradually to the other end of the tube, the heat being increased very considerably at the close of the operation, so as to fuse the chromate of lead. By this means, the whole carbon of the cast-iron is converted into carbonic acid, which is condensed by the solution of potash. It is unnecessary to give the details of this process here, as they are all included in the account of the operation of organic analysis.

(For the methods of estimating carbonic acid when contained in aqueous solution see "Analysis of mineral Waters.")

XI. WATER.

Estimation of water as contained in hydrates. — The ordinary method of determining the amount of water contained in a substance consists in expelling the water by the application of heat, and estimating it by the loss in weight which the substance then experiences. The degree of heat requisite varies according to the facility with which the water is expelled, and also with the nature of the hydrate under examination. Either a platinum or porcelain crucible may be employed in this operation.

In general, this simple process gives a satisfactory result, but a large number of bodies containing water exist, which can experience a change in weight on being heated, from circumstances entirely unconnected with the presence of water. For such compounds, the process must be modified. If the hydrate to be analyzed possesses the property of uniting with oxygen at a high temperature, to obtain the water, the sub-

stance is heated in an atmosphere of some gas which exerts no action on it, such as carbonic acid or hydrogen; a current of which, previously dried by passing through a chloride of calcium tube, is sustained during the whole operation. Such an apparatus as that figured at page 160. may be used; and the water given off by the substance may be collected and weighed in a small chloride of calcium tube attached to the posterior extremity of the tube *c*. At the end of the process, and when the substance is cold, air is admitted into the tube, to bring it to the same state as when weighed before the experiment. The loss in weight indicates the amount of water.

When the substance to be analyzed contains an ammoniacal salt, that is expelled, together with the water, by ignition. The entire loss in weight being determined, the amount of ammonia contained in the substance must then be ascertained by a separate experiment; the corresponding quantity of ammoniacal salt is calculated and deducted from the whole loss, to obtain the amount of water.

In determining the proportion of water in those salts which lose the whole or a portion of their acid at a high temperature, the substance should be intimately mixed with a known weight of a strong base which can retain the acid, forming a salt which is not decomposed at the temperature at which water is given off. Protoxide of lead is the base generally employed for this purpose, of which oxide, from four to six times the weight of the substance may be taken. On heating, water alone is expelled, unless the compound contains an ammoniacal salt, when free ammonia is also given off.

In a case where the hydrate to be analyzed is that of an organic body, if the whole of the water is not removed by heating at a temperature lower than that at which the organic matter suffers decomposition, recourse must be had to an ultimate organic analysis, provided the substance possesses a definite constitution. The whole of the water is then obtained, condensed in the chloride of calcium tube (see the account of the operation), but with it an additional

quantity proceeding from the combustion of the hydrogen of the organic substance; the proportion of which in relation to the carbon being known, the quantity of water it should produce is calculated and deducted from the whole amount obtained. The remainder is the quantity which existed in the substance as water. In the determination of combined water, care should be taken that the body is entirely free from hygrometric moisture, and if in a crystallized state, that it contains no water lodged mechanically between the plates of the crystals. If such is suspected, the crystals must be powdered and carefully dried by heating a few degrees above the ordinary temperature.

As almost all hydrates of definite constitution evolve different quantities of water at different elevations of temperature, it is advisable to commence by heating the substance gently, and to increase the temperature progressively, weighing at each increase. This is especially necessary in all accurate investigations of hydrates of salts or of organic bodies: after drying as much as possible in the open air, the substance may be kept in a confined portion of air, supported over a surface of oil of vitriol; then in vacuo, over oil of vitriol; afterwards it may be heated to 212° by a water-bath, or higher, if necessary, by means of a boiling saturated solution of some salt, such as chloride of sodium or nitre.

The proportion of water contained in inorganic hydrates which do not part with their water by being heated is estimated as the loss, the weights of all the other constituents of the substance being first determined.

ANALYSIS OF MINERAL WATERS.

(1.) *Saline, Chalybeate, and Carbonated.*

Although the qualitative analysis of a mineral water may exhibit the presence of some bodies of rare occurrence, such as iodine, bromine, strontian, lithia, ammonia, &c., the quantities of these are, in general, so small as to render it worthless, except in special investigations, to attempt to estimate

their weight. The following directions for the quantitative analysis of mineral waters, will not, therefore, be complicated with details of the modes of estimating such bodies (for which I refer elsewhere), but comprise the means of estimating those only which are of ordinary occurrence. In saline, chalybeate, and carbonated waters, the common constituents are : — *lime, oxide of iron, magnesia, soda, manganese, silica, sulphuric acid, chlorine, and carbonic acid.*

The operations to be performed in the estimation of the fixed constituents are : —

1. Evaporation to dryness to ascertain the whole amount of saline matter.
2. Estimation of the magnesia, lime, oxide of iron, oxide of manganese, and silica, contained in the portion of the residue of evaporation which is insoluble in water.
3. Estimation of the lime, magnesia, and oxide of iron, contained in the portion of the residue of evaporation which is soluble in water.
4. Estimation of the whole amount of lime, magnesia, oxide of iron, and silica.
5. Estimation of the whole amount of sulphuric acid.
6. Estimation of the whole amount of hydrochloric acid (chlorine).
7. Estimation of the whole amount of chlorides.
8. Estimation of the whole amount of chloride of sodium.

1. To obtain the entire amount of saline matter contained in the water, a known quantity is evaporated to dryness in a porcelain bason, or what is better, in a weighed platinum capsule. The evaporation should be conducted below 212° to prevent ebullition, and consequent loss from spiriting; but when the residue is quite dry, the heat should be gradually increased to 300° or 400° Fahr. The weight of the dry residue affords a check on the subsequent determinations.

During evaporation, carbonic acid gas may probably be disengaged, and a precipitate formed at the same time; the latter may consist of carbonate of lime, carbonate of magnesia, and oxide of iron. These bodies were formerly held in solution by the carbonic acid, and are consequently pre-

precipitated when the latter is expelled. On treating the dry residue of the evaporation with water, this precipitate, together with some silica, and perhaps oxide of manganese, remains undissolved. The solution in water may also contain portions of lime, magnesia and oxide of iron, but these portions did not originally exist in the water as carbonates.

2. The second operation consists in the estimation of the lime, magnesia, oxide of iron, and silica contained in the precipitate formed on ebullition. The dry residue of evaporation is treated with water, and as much of the precipitate as can be easily removed is thrown on a filter and washed. What remains of the precipitate attached to the sides of the capsule, after being washed, is dissolved in hydrochloric acid, and the acid liquid passed through the filter to form a solution of the whole precipitate. If an insoluble residue exists, it is *silica*, which should be washed, ignited, and weighed. The solution of the precipitate in hydrochloric acid may contain lime, magnesia, oxide of iron, and manganese. Its analysis is performed according to the following table:—

TREATMENT OF THE SOLUTION IN HYDROCHLORIC ACID OF THE PRECIPITATE FORMED DURING EBULLITION.

Add a few drops of nitric acid to the solution to peroxidize iron and apply heat. Neutralize the free acid with ammonia; add, first, muriate of ammonia, and afterwards hydrosulphate of ammonia.

The precipitate is sulphuret of iron and sulphuret of manganese; filter, wash, and dissolve it in hydrochloric acid: heat the solution to expel sulphuretted hydrogen, and saturate in the cold with carbonate of barytes.

The precipitate is oxide of iron mixed with carbonate of barytes. Filter, dissolve it in hydrochloric acid, and add ammonia to the solution. Peroxide of iron is precipitated and may be dried and weighed.

The solution contains lime and magnesia (hydrosulphate of ammonia). Add hydrochloric acid, boil to expel sulphuretted hydrogen, and filter: add to the solution first a slight excess of ammonia, and then oxalate of ammonia. Filter.

Oxalate of lime is precipitated. Convert it into carbonate by ignition, and weigh (page 138.) The solution contains magnesia; add phosphate of soda and ammonia. Convert the precipitated ammonio-phosphate of magnesia into *phosphate of magnesia* by ignition, and weigh.

3. The estimation of the lime, magnesia, and iron contained in the soluble portion of the residue of evaporation is performed in the same manner, pure ammonia being substituted for hydrosulphate of ammonia to precipitate oxide of iron.

4. The fourth operation is merely confirmatory of the last two, having for object the determination of the entire amounts of lime, magnesia, iron, manganese, and silica contained in the water. A little nitric acid is added to a known volume of the water, which is evaporated carefully to dryness. On treating the residue with very dilute nitric acid, silica remains undissolved, which may be collected and weighed, and the solution is proceeded with according to the table.

5. The fifth operation is to determine the whole amount of sulphuric acid. Add a little hydrochloric acid to a known volume of the water, and estimate the sulphuric acid as sulphate of barytes.

6. To another portion of the water add an excess of nitric acid, and precipitate the chlorine by nitrate of silver. Weigh the chloride of silver, and calculate its amount of chlorine.

7. To determine the whole amount of chlorides, boil a portion of the water, and filter from the precipitate produced thereby. To the filtered liquid add muriate of ammonia, and evaporate to dryness by a water-bath, the evaporation being commenced in a porcelain bason and terminated in a weighed platinum capsule. When the saline residue is quite dry, the heat should be increased so as to bring it to a state of fusion, after which it may be weighed. The residue consists of a mixture of the chlorides with the sulphate contained in the water, the latter being either sulphate of soda or sulphate of lime. Having previously determined which of these sulphates exists by the qualitative analysis of the water, the amount of the *sulphate* is calculated from the quantity of *sulphuric acid* before obtained. On deducting the weight of the sulphate from that of the saline residue the weight of the chlorides is determined. The addition of muriate of ammonia is necessary to prevent decomposition of chloride of

magnesium by ignition: the hydrated chloride gives off hydrochloric acid, and leaves magnesia when heated to redness, but the double chloride of magnesium and ammonium, which is formed when muriate of ammonia is brought into contact with chloride of magnesium, gives off muriate of ammonia, and leaves anhydrous chloride of magnesium, which undergoes no change by being heated.

8. The amount of chloride of sodium is ascertained in the following manner: — A known weight of the water is concentrated by evaporation, and filtered from the precipitated earthy carbonates.

Barytes-water is added to the filtered solution.

The precipitate contains magnesia, lime, and sulphate of barytes. Filter.

The solution contains lime, barytes, soda, and chloride of sodium. Add to it a solution of carbonate of ammonia.

The precipitate contains carbonate of barytes and carbonate of lime.

The solution affords, when evaporated to dryness and the residue calcined, a mixture of chloride of sodium and carbonate of soda. Dissolve this residue in nitric acid, and estimate the chlorine as chloride of silver. From the weight of the chloride of silver that of the chloride of sodium is calculated.

If the analysis is exact, the chlorine contained in the chloride of sodium deducted from the whole amount of chlorine, should, in general, leave a quantity equivalent to the calcium and magnesium which remain in solution after boiling.

Estimation of the volatile constituents of saline waters. — The volatile constituents of saline waters are carbonic acid, and free oxygen and nitrogen. Carbonic acid is one of the most important ingredients of mineral waters. It may exist either in the free state, or united with lime, magnesia, oxide of iron, or soda. Several methods of determining the proportion of carbonic acid have been devised, the one most generally practised, but perhaps the least accurate of which, consists

in boiling a known quantity of the water in a flask which has an exit tube terminating under an inverted graduated bell-jar of mercury. In this jar the gases evolved by ebullition are collected. Potash being introduced into the gas, the carbonic acid is absorbed, from which absorption, the requisite corrections for pressure, temperature, and humidity being attended to, the amount of carbonic acid capable of expulsion from the water by boiling is estimated. The precipitate formed during ebullition contains a certain amount of carbonic acid, which amount may be calculated after the composition of the precipitate is ascertained by analysis, and added to that collected as gas, to obtain the entire quantity. Instead of collecting the gas over mercury, the exit tube may be made to dip into a solution composed of a mixture of chloride of calcium and ammonia, when all the carbonic acid is precipitated in the form of carbonate of lime, which may be weighed.

A simpler and more accurate method of determining the entire amount of carbonic acid in a mineral water is the following:—A known quantity of the water, which need not be very considerable, fresh from the spring, is mixed with caustic ammonia. The alkali immediately combines with the free carbonic acid to form carbonate of ammonia; at the same time, all the lime and a portion of the magnesia of the water are precipitated as carbonates, together with oxide of iron and a little silica. It rarely happens that the water contains a sufficient quantity of lime salts to unite with all the carbonic acid of the carbonate of ammonia, so that to obtain all the carbonic acid in the form of an insoluble carbonate, a solution of chloride of calcium should be added. After the liquid has been boiled to enable the precipitate to subside, it is filtered, with as little exposure to the air as possible, that no additional carbonate of lime may be formed through absorption of carbonic acid from the atmosphere. While the precipitate is yet moist, the filter is folded and passed up into an inverted graduated jar, at the mercurial trough, the jar being filled with mercury. Dilute hydrochloric acid, previously saturated with carbonic acid by

dissolving a fragment of chalk in it, is then introduced into the jar by means of a bent pipette. When the acid comes into contact with the carbonates in the filter, the latter are decomposed, with evolution of carbonic acid gas, equal in quantity to the entire amount originally contained in the water operated on. According to this method, the capacity of the mercurial jar should be nearly double the volume of the water taken for analysis; but as a single jar of sufficient capacity can rarely be procured, the same result is obtained without its use in the following manner:— The precipitated mixed carbonates are collected on a weighed filter, and when perfectly washed, are dried at 212° and weighed with the filter. The entire amount of the carbonates being thus known, small weighed portions are taken successively and decomposed by hydrochloric acid in the above manner, but in a smaller mercurial jar. The mean quantity of carbonic acid afforded by five or six such experiments is taken, and the proportion calculated for the entire amount. By this means we obtain the absolute quantity of carbonic acid contained in the water, that is, both that which exists in a free state and that which exists in the earthy carbonates. To know how much is contained in each of these states, another portion of the water is taken, equal in bulk to that before operated on, and boiled for half an hour to precipitate the carbonates, which are collected on a filter. When this precipitate is decomposed by hydrochloric acid in the mercurial jar in the manner described, it affords the quantity of carbonic acid existing in the carbonates only, which, deducted from the entire amount previously obtained, of course leaves the proportion of carbonic acid existing in the free state.

A much simpler means of determining the amount of carbonic acid in the precipitated carbonates would be by the use of the little apparatus for the analysis of carbonates described page 233. I am not aware, however, if this method has ever been practised.

Besides carbonic acid, all waters which are exposed to the atmosphere hold in solution free *oxygen* and *nitrogen*. To

determine the proportions of these gases, the water is boiled in a flask, to the cork of which an exit tube is adapted, leading into the mercurial trough, and terminating under an inverted jar filled with mercury. In this jar the evolved gases are collected. The flask and tube should be completely filled with the water for examination. When all the gas is collected in the jar, introduce into it, first, a stick of potash, to absorb carbonic acid, and into the remaining air, or a portion of it, a stick of phosphorus. The latter, in a short time, absorbs all the oxygen, leaving a residue of pure nitrogen.

(2.) *Alkaline Waters.*

As bicarbonate of potash rarely enters into the composition of a mineral water, if it is ascertained that an alkaline bicarbonate does exist, the base may be considered to be soda. When other soluble salts are present, the accurate estimation of the alkali requires operations of some difficulty. To obtain the alkali, first, a known bulk of the water, previously well boiled, is slightly acidified by nitric acid, and its amount of chlorine determined as chloride of silver. Another portion of the boiled water, also a known quantity, is then mixed with some muriate of ammonia, and evaporated carefully to dryness, the evaporation being terminated in a platinum capsule heated by a water-bath. By boiling, the carbonate of soda contained in the water, and the muriate of ammonia added, suffer mutual decomposition, affording carbonate of ammonia and chloride of sodium: instead of carbonate of soda, therefore, the residue of the evaporation contains the equivalent quantity of chloride of sodium. After being brought to a state of perfect dryness in the water-bath, the residue is ignited at a red heat, dissolved in water, and the amount of chlorine contained in the solution estimated as chloride of silver. Now the excess in the amount of chlorine here obtained over that before determined as pre-existing in the water is the quantity of chlorine contained in the chloride of sodium which was formed from the decomposition of the alkaline carbonate, from which, there-

fore, the amount of the latter can be obtained by calculation.

The carbonic acid and other ingredients of alkaline waters are determined in the same manner as in saline waters.

(3.) *Sulphureous Waters.*

In a sulphureous water the most important ingredient is sulphur, which element may exist both in the state of free sulphuretted hydrogen, and as a soluble sulphuret. The proportion of sulphur may be obtained by the following process, first described by Wöhler: — To a known quantity of the water is added a solution of nitrate of silver, with which a sufficient quantity of caustic ammonia has been previously mixed to prevent the precipitation of any chloride of silver, the latter compound being soluble in ammonia. The precipitate which falls, consisting of a mixture of sulphuret of silver and earthy carbonates, is collected on a weighed filter, and washed, first with ammonia, next with dilute acetic acid to decompose the carbonates, and lastly, with pure water. The sulphuret of silver is dried at 212° , until it no longer loses weight, and from its amount that of the sulphur it contains is calculated.

By this operation the *whole amount of sulphur* contained in the water is ascertained. To know how much exists as free sulphuretted hydrogen, and how much as a soluble sulphuret, another operation is necessary. A known quantity of the water is boiled in an open vessel until the odour of sulphuretted hydrogen is no longer perceptible. After being boiled, the liquid is filtered, and the sulphur it now contains estimated in the same manner as before. By deducting the quantity thus obtained from the entire amount, that contained as sulphuretted hydrogen, which was expelled by ebullition, is ascertained.

When free iodine is brought into contact with sulphuretted hydrogen, or a soluble sulphuret, the latter is decomposed, affording hydriodic acid, or an iodide, with precipitation of free sulphur. On this decomposition, M. Pasquier

has lately founded an elegant method of ascertaining the presence and quantity of sulphur in a mineral water, by observing what amount of free iodine is taken up, or caused to enter into a state of combination, by a known quantity of the water. The process is as follows :— An alcoholic solution of iodine, conveniently diluted and of known strength, is carefully dropped from a graduated pipette into a known weight of the water, mixed with a little starch, until the mixture commences to assume a blue tint. So long as any sulphuretted hydrogen or soluble sulphuret is present, it is decomposed by the free iodine, which passes into a state of combination, so that the peculiar effect of iodine on starch (that of rendering it blue) is not exhibited until an excess has been added. From the quantity of iodine added, that of the sulphur which it represents may be calculated.

Ingenuous and elegant as this process is, it is exposed, in its present form, to a source of error of too great importance to be overlooked. If, at first, alcohol does form, strictly, a *solution* of iodine, the latter is not very slow to react on the elements of the alcohol, forming, among other products, hydriodic acid. There is, hence, a loss of free iodine, and the solution is no longer normal. If, however, instead of alcohol, a solution of chloride of potassium or chloride of sodium is employed to form the solution of iodine, the process might be made to afford results of great accuracy.

The fixed constituents of sulphureous waters are determined in the same manner as those of saline waters.

DETERMINATION OF THE DEGREE OF HARDNESS OF WATERS.

An operation of much more general application than the complete analysis of a mineral water is a simple and ready means of determining what is called the *hardness of waters*, or in other words, the quantity of earthy bases present in solution ; for which, an excellent process, affording numerical results of considerable nicety, has been recently contrived by

Dr. Clark of Aberdeen. The process consists essentially in adding a *normal solution of soap* in proof spirit, from a graduated vessel, to a known quantity of the water, until a lather is produced on agitation. As the lather is formed only when the soap is in excess, and as that cannot be the case until all the earths are precipitated, the quantity of solution of soap necessary to produce the lather is exactly proportional to the quantity of earthy bases present, that is, to the hardness of the water. The following are the details of the process.

For purposes of comparison, Dr. Clark employs "standard solutions" of chloride of calcium in distilled water: these serve to prepare the solution of soap, called the "soap test," of known strength, and also to compare or refer the degree of hardness of the water under examination. To prepare these standard solutions dissolve 16 grains of pure carbonate of lime in a small quantity of hydrochloric acid, carefully avoiding loss from effervescence; evaporate the solution to dryness, redissolve the residue in water, and again evaporate. Repeat the solution and evaporation until a neutral solution is obtained. The quantity of chloride of calcium thus obtained, affords, by being dissolved in a gallon of distilled water, what is named "the standard solution of sixteen degrees of hardness," from which all the other standard solutions are prepared. A mixture of 1 measure of this solution with 15 of distilled water constitutes the standard solution of one degree of hardness: 2 measures mixed with 14 of water, and 3 measures mixed with 13 of water, form respectively solutions of two and three degrees of hardness. In a similar manner, solutions are prepared up to sixteen degrees.

The soap preferred, in the preparation of the soap test, is that known technically in London as "Hawes' white curd." As that is not always constant in composition, it is necessary, in order to prepare a solution of known strength, to make a preliminary trial on a small scale, to ascertain approximately what quantity of soap should be dissolved in the spirit. For this purpose a small quantity of a solution is

made in the proportion of one ounce avoirdupois of soap to one gallon of proof spirit, and the actual strength of this solution is determined as follows: — “Let 100 test measures” (a single test measure employed by Dr. Clark is the $\frac{1}{7000}$ th part of the imperial gallon, that is, a measure equal to ten grains of water,) “of the standard solution of sixteen degrees of hardness be put into a phial capable of holding twice this quantity. Into the water in this phial let the prepared trial solution of soap be gradually poured from a measure graduated into test measures; let the mixture be shaken after each addition of the solution of soap, and let the operation be continued in this manner until such time as a lather will be formed of a sufficient consistence to remain for five minutes all over the surface of the water, when the phial is placed on its side. The number of test measures thus required will be either 32, or more or less than 32. If the number prove to be 32, then more solution of soap may be made in the same proportions as in the trial solution, and the solution so made will be the soap test.” If the number be more or less than 32, then proportionally more or less of the soap must be employed in preparing the soap test. “But in each case the soap test, after being prepared, should be carefully verified by a trial in the manner described, so as to ascertain whether 100 test measures of the standard solution of sixteen degrees of hardness will afford a lather with exactly 32 measures of the soap test. If not, an adjustment must be made.”*

The test solution thus prepared is applied in the same manner: it is gradually added to 100 test measures (1000 grains) of the water, until, on agitation, a lather of sufficient consistence is formed to remain for five minutes when the phial is placed on its side. The water is said to correspond in degree of hardness with that standard solution which requires the same number of test measures of soap solution as itself. Thus, if 32 are required, it is said to be of sixteen degrees of hardness, 32 being the number required

* Specification by Professor Clark of a new mode of rendering certain waters less impure and hard, &c., pages 4. and 5.

by the standard solution of sixteen degrees. But if to form the lather 100 test measures of the water require a number of measures of soap solution different from that required by any standard solution (being less than 32), its degree of hardness is stated in fractions obtained by computation from the difference between the number of measures required by the standard solution next above to that next below it. "Thus, in forming the lather, if 100 test measures of the standard solution of eleven degrees were found to take $1\frac{8}{10}$ test measures of soap test, more than what 100 test measures of the standard solution of ten degrees would require," (for each degree of hardness is equivalent to less than 2 measures, although 32 measures indicate sixteen degrees,) "and if 100 test measures of the water under trial for hardness require $\frac{9}{10}$ of a test measure over and above what the same standard solution of ten degrees would require, then the water under trial would be said to be $10\frac{5}{10}$ degrees of hardness. This fraction is obtained by proportion thus: "—

$$\begin{array}{ccccc}
 \text{Differences of soap} & & & & \text{Differences in degrees} \\
 \text{test.} & & & & \text{of hardness.} \\
 \hline
 \overbrace{1\frac{8}{10} : \frac{9}{10}} & :: & \overbrace{1 : 10\frac{5}{10}}
 \end{array}$$

If 32 measures of soap solution are insufficient to produce the lather with 100 measures of the water, 100 measures of distilled water are added, and the addition of the soap solution is continued as before. If at 60 or a lower number the lather is obtained, a final trial is made on a mixture of 100 measures of the water for examination with 100 of distilled water, the number of measures required of the test solution is divided by 2, and the degree of standard solution to which this number corresponds is observed: the double of this degree is the hardness of the water. If, for instance, one half the number of measures of soap test required corresponds to $10\frac{5}{10}$ degrees of hardness, the hardness of the water under examination will then be twice that number, or 21.

If 60 test measures are insufficient to produce the lather,

another 100 measures of distilled water are added, and the addition of soap continued as before to 90 measures: when, if necessary, another 100 measures of distilled water are added, and the addition of soap continued. A final trial must always be made on a mixture of 100 measures of the water for examination with as much distilled water as was used in the preliminary experiment; that is, 100 measures for every 30 of soap test above 30. The number of measures of soap test required is reduced by a simple divisor to a number below 32; the corresponding standard solution is obtained, and the degree of hardness of the latter is multiplied by the number before used as the divisor. This gives the degree of hardness of the water for trial. If, for example, the number of measures of soap test employed gives, when divided by 3, a number which corresponds to $10\frac{5}{10}$ degrees of the standard solutions, the hardness of the water is three times that number, or $30\frac{9}{10}$.

From the way in which the standard solutions are prepared, each degree represents a quantity of chloride of calcium equivalent to 1 grain of carbonate of lime in a gallon of water, the solution of sixteen degrees being prepared by dissolving 16 grains of carbonate of lime, by means of hydrochloric acid, in 1 gallon of water; and since, in ordinary waters, the hardening impurity is almost solely carbonate of lime held in solution by free carbonic acid, the degree of hardness of the water for examination generally expresses the number of grains per gallon of carbonate of lime contained in the water.

This process for ascertaining the amount of hardening impurity in ordinary waters is more simple in execution than might be expected from its description.

SECTION VI.

ORGANIC ANALYSIS.

Most organic bodies contain only three elements, namely, carbon, hydrogen and oxygen; in addition to these, a comparatively small number of organic substances possess nitrogen, while still fewer, only carbon and hydrogen, carbon and oxygen, or carbon and nitrogen. To determine the proportions of these elements is the object of organic analysis.

- (1.) *Carbon* is determined by conversion into *carbonic acid*, which is condensed in a weighed solution of caustic potash.
- (2.) *Hydrogen* is determined by conversion into *water*, which is collected and weighed in a chloride of calcium tube.
- (3.) *Nitrogen* is determined in two ways; either by collecting it in the *free state as gas*, or by converting it into *ammonia*, and forming the ammonio-chloride of platinum.
- (4.) *Oxygen* is always determined as *loss* on the original weight of the substance.

For the conversion of carbon into carbonic acid and of hydrogen into water, and to obtain nitrogen in a free state, the organic substance is subjected to a process of combustion by oxygen. It may be burned in pure oxygen gas, as in Dr. Prout's process, but in the ordinary methods, the organic substance is mixed with some solid body capable of yielding oxygen to it on the application of heat. In the processes here described, the material employed for that purpose is either pure *oxide of copper*, *oxide of copper mixed with chloride of potash*, or pure *chromate of lead*.

Whichever of these three is used, as the hydrogen of the organic substance is to be weighed in the form of water, obviously every pains must be taken to insure the absence of all hygrometric moisture before the weight of the substance is taken, that no water but what is actually produced in the

combustion may be collected and weighed. One way of drying the substance is by means of an ordinary water-bath, the substance being contained in a shallow bason placed over a deeper one containing boiling water, or a saline solution, if the temperature of 212° is insufficient. Instead of an open bason, the substance may be placed in a U-shaped tube, one extremity of which is connected with a small chloride of calcium tube, and the other with a suction bottle similar to *a fig. 22.* page 232., by means of which air is slowly drawn through the tube, the air being previously rendered perfectly dry by passing through the tube containing chloride of calcium at the opposite extremity of the bent tube. This is a very convenient apparatus to use when it is required to determine the quantity of water lost by the substance at the temperature of the bath, in which case another chloride of calcium tube is attached, to collect the water given off, being placed between the bent tube and the suction bottle.

As the suction bottle referred to might be difficult to procure, an efficient substitute for it may be made out of an ordinary single-mouthed bottle, of sufficient dimensions, as follows:— Three perforations are made through the cork of the bottle, in one of which terminates a small tube, in connection with the U-shaped tube containing the substance to be dried. Through another perforation, a syphon tube is passed, reaching to the bottom of the bottle, the longest limb of the syphon being outside of the bottle. The use of the third opening is to renew the supply of water in the bottle without taking out the cork, and is furnished with an air-tight plug. Water being introduced into the bottle, and the syphon completely filled, the water in the bottle is withdrawn, air entering through the U-shaped tube at the same time to supply the place of the water.

To ascertain if the substance is dry, a small portion is heated in a narrow test tube to the highest temperature which the body will support without decomposition; if a mere trace of water is present, it is discoverable by the moisture which condenses on the side of the tube.

When perfectly dry, a portion of the substance is weighed

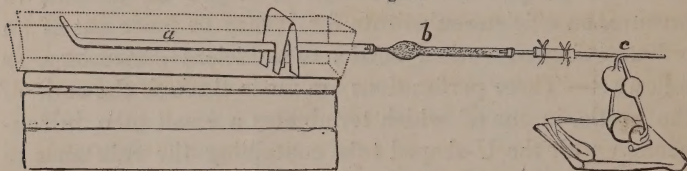
out for analysis: in general, from seven to nine grains may be taken. While being weighed, it is most conveniently contained in a glass tube sealed at one end, about two inches in length, and from one fourth to one third of an inch in diameter.

The various steps in the combustion of a pulverizable and non-volatile substance by oxide of copper are as follow: —

Preparation of the oxide of copper. — The oxide is best prepared from the nitrate by calcination in an earthenware crucible at a dull red heat, until ruddy fumes are no longer evolved, and no green particles are perceived mixed with the black oxide. The nitrate itself is made by the action of nitric acid on perfectly clean pieces of sheet copper. During calcination the mass should be stirred with an iron or a copper rod.

Apparatus. — The apparatus employed in the combustion is exhibited in the annexed cut: *a* is the *combustion tube*, in

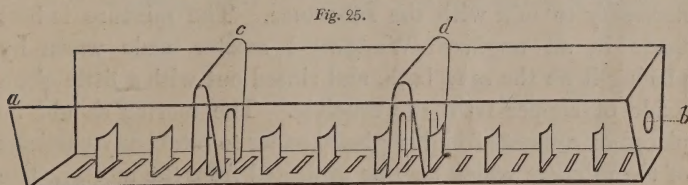
Fig. 24.



which is contained the mixture of oxide of copper with the organic substance to be analyzed. The best tubes employed are those made of the hard Bohemian potash glass. In general, the tube should be about fourteen or fifteen inches long, and about four tenths of an inch in diameter; one extremity is drawn out to a point and sealed as shown in the figure, and the other is open and fused so as to receive a cork without danger of splitting. The tube should be cleaned by washing with water, and then dried perfectly by heating and sucking air out at the same time by a long narrow tube introduced to the end of the combustion tube. By means of a perforated cork, the combustion tube is connected with the tube *b*, filled with chloride of calcium which has been strongly dried but not fused. Large fragments of

that salt are placed in the bulb, and coarse powder in the tube: at each end a little cotton wool is placed to prevent any of the chloride of calcium from falling out. The use of this tube is to retain all the water produced in the combustion. Connected with the chloride of calcium tube by means of a caoutchouc joint is the potash apparatus *c*, the use of which is to absorb the carbonic acid. It consists of a tube on which five bulbs are blown, three at the bottom, the middle of which is the largest, and one on each of the sides; of the side bulbs one must be larger than the middle of the three bottom bulbs.* The extremity of this apparatus in connection with the large bulb is attached to the chloride of calcium tube. The apparatus is filled with the solution of potash by sucking with the mouth at one end, while the other end dips into a wine glass containing the liquid. It is filled in such a manner, that each of the three lower bulbs contains a small bubble of air, none of the liquid being contained in the upper bulbs. The alkaline solution is of proper strength when it possesses a specific gravity between 1.25 and 1.27.

The combustion tube is heated in a furnace (*fig. 25.*)



made of sheet iron, from 22 to 24 inches long, 3 inches high, $4\frac{1}{2}$ inches wide at top, and 3 inches wide at bottom. One end of the furnace is open *a*, the other end has a front in which is a round aperture *b* to admit the combustion tube. Across the bottom are apertures or slits, serving to form a grate; they are about half an inch distant from each other, and each aperture is one inch and a half long, and a quarter of an inch wide. At every second aperture, a support is

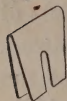
* This, with all the other apparatus employed in organic analysis, may be procured of Messrs. R. Griffin and Co. of Glasgow.

placed for the combustion tube made of stout sheet iron.



One of these supports is represented in *fig. 26*. To confine the fire to particular portions of the tube, two screens *c* and *d* (*fig. 25.*) are employed, made of sheet iron; a figure of one of these is annexed

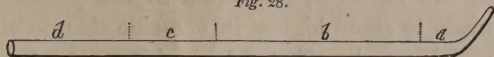
Fig. 27.



(*fig. 27.*). The furnace rests on a paving stone or a couple of flat bricks, the front projecting a little so that the first slit is open, but all the others closed. One end of the furnace is raised a little, as shown in *fig. 24.*, so that the water which condenses in the anterior part of the chloride of calcium tube may flow into the bulb.

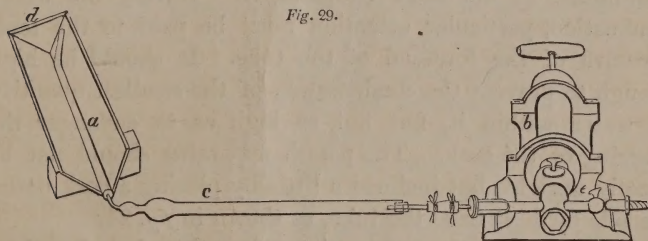
Manipulations. — The whole of the apparatus being complete, and the weights of the potash apparatus and chloride of calcium tube taken, the operator may proceed to mix the substance to be analyzed with oxide of copper. Immediately before being used, the oxide of copper is heated to dull redness in an earthen crucible, and when cooled down to about 300° or 400° , enough is introduced into the combustion tube to fill the latter a little more than one half. The object of introducing the oxide is to have a measure of the quantity necessary to mix with the substance. The mixture is best made in an unglazed Wedgwood mortar made warm by placing it on the sand bath, and rinsed out with a little clean oxide of copper from the crucible. The mortar should be placed on a sheet of highly glazed paper to retain any particles of the mixture which may be projected from the mortar. The substance to be analyzed is transferred from the tube in which it is weighed to the mortar; and the tube with the adhering particles of the substance, is immediately placed on the pan of the balance or some place of security to be weighed at a convenient opportunity. The oxide of copper in the combustion tube is now poured gradually into the mortar, leaving about half an inch at the extremity of the tube; the oxide is mixed gently but perfectly with the substance, and the mixture is introduced into the tube. The mortar is then rinsed out two or three times with oxide of copper from the crucible, and the rinsings are introduced into the tube, being

placed above the mixture; above the rinsings is sufficient pure oxide of copper placed to reach within one inch of the open end of the tube. In *fig. 28.* *a* represents pure oxide,

Fig. 28.

b the mixture, *c* the rinsings of the mortar, and *d* pure oxide.

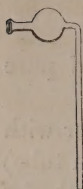
The tube is now closed by a cork, which (together with the perforated cork to receive the chloride of calcium tube) has been dried by heating in a bason on the sand bath for some hours, and the tube is gently tapped on a table, being held in a horizontal position, so that a clear space is seen above the oxide throughout the whole length of the tube. While the mixture is being made, both the oxide of copper and the substance absorb a quantity of water from the air, amounting to as much as 0.3 grain (equivalent to 0.5 per cent of hydrogen), to get rid of which the mixture is dried in vacuo at a somewhat elevated temperature. The apparatus figured in the annexed cut is employed for that purpose. The combustion tube *a* (*fig. 29.*) is connected with an ex-

Fig. 29.

hausting syringe *b*, a long chloride of calcium tube *c* intervening: the combustion tube itself is placed in a wooden trough *d*, and surrounded with sand of a temperature about 250° Fahr. On exhausting the air of the apparatus by the syringe, moisture is withdrawn from the combustion tube: air, dried by passing through the chloride of calcium tube *c*, is re-admitted by opening the stop-cock *e*, and again withdrawn. The exhaustion and admission of air having been

repeated ten or a dozen times *, the combustion tube may be connected with the potash apparatus and chloride of calcium tube, arranged as in *fig. 24*. To ascertain whether all the connections are air-tight, the liquid in the potash bulbs is

Fig. 30.



made to stand higher in one side than in the other, which may be effected by sucking out a few bubbles of air from the apparatus by the suction tube (*fig. 30.*), after which, the potash ley rises to supply the place of the air withdrawn. If the connections are imperfect, the liquid will return to the same level in both limbs, but not otherwise.

The apparatus being proved air-tight, the portion *d* (*fig. 28.*) of the tube which contains pure oxide of copper, is now surrounded with red-hot charcoal, the heat being prevented from spreading by the screen (*fig. 27.*). When that part is heated to redness, the screen is gradually advanced to the posterior end of the tube, moving from one half of an inch to one inch at a time, according to the rapidity of the evolution of gas, as perceived in the potash apparatus. As the screen is advanced, each portion of the tube must be quickly raised to a red heat, the anterior portion being all along maintained at the same temperature. During the whole combustion, particular attention must be paid to the temperature of the fore-end of the tube. It should be high enough to prevent the condensation of the smallest quantity of water within it, but not so high as to endanger the charring of the cork. The potash apparatus should not be exactly upright, but inclined a little by placing a cork under the side farthest from the tube, as shown in *fig. 24*.

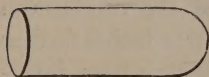
At the end of the combustion, when the whole length of the tube is heated, the heat is increased by raising one side of the furnace a little to admit air through all the slits, and also by fanning from above with a piece of pasteboard. When the evolution of gas ceases or becomes very slow, the potash apparatus is restored to its horizontal position, and

* At this period of the operation a convenient opportunity offers for weighing the tube in which the substance was weighed.

the ley is allowed to rise in the bulb next the chloride of calcium tube (by absorbing the carbonic acid gas), until it reaches such a height that air can enter through the three lower bulbs. The charcoal is then removed from the sealed end of the combustion tube, the point of which is divided by a small pair of pliers. Air then enters the apparatus, from the potash ley falling to the same level in both sides of the apparatus, provided there is no stoppage in the combustion tube. The suction tube (*fig. 30.*) is now adapted to the end of the potash apparatus, and a volume of air equal to about twice the capacity of the entire apparatus is slowly sucked through it. The current of air carries forward all the watery vapour and carbonic acid remaining in the apparatus, which are absorbed by the chloride of calcium and the potash. The potash apparatus and the chloride of calcium tube may now be disconnected and weighed to ascertain the increase in weight each has sustained.

Combustion of volatile organic substances by oxide of copper. — The combustion of a volatile substance must obviously be conducted in a manner somewhat different from the preceding. If the substance is solid at common temperatures, and fusible, such as naphthaline or benzoic acid,

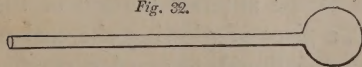
Fig. 31.



it is fused and weighed in a tube of the size and shape represented in *fig. 31.*, and both tube and substance are introduced into the combustion tube, being placed

about one inch and a half from the closed end of the latter. If the substance is liquid at common temperatures, it is weighed in one, two, or three small glass bulbs, one of which

Fig. 32.



is shown of the proper size in *fig. 32.* To fill the bulb, it is heated so

as to expel a portion of the air within, and while hot, the open end of the neck is dipped under the surface of the liquid to be introduced. When a little of the liquid has entered through the contraction of the heated air, the bulb is again gently heated, on which more air is expelled; and on dipping the open end again into the liquid, an additional

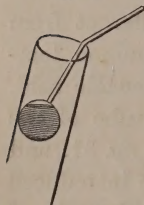
quantity of the latter enters. Each bulb should be about three fourths full. The liquid being introduced, the point is sealed, and the weight of the bulb and liquid taken; the weight of the empty bulb (previously ascertained) deducted from its present weight gives that of the liquid introduced.

Before being used, the oxide of copper is heated to red-
 Fig. 33. ness in an earthen crucible, and transferred while hot to the bulb and tube, or flask (*fig. 33.*) in which it is allowed to cool, the vessel being carefully closed with a well-dried cork. When the oxide is quite cold, the flask is uncorked, its mouth is applied to the open end of the combustion tube, and one inch or one inch and a half of oxide is shaken in. If the substance for analysis is a solid, the small tube containing it is now dropped in, with its sealed end undermost, and



the combustion tube is filled with oxide of copper to about one inch of its extremity. If the substance is a liquid, the bulb in which it is contained is scratched by a file at the middle of its neck, which is broken across when introduced into the combustion tube, as represented in *fig. 34.*, both bulb and neck being allowed to slide down the tube. If two bulbs are used, the first is placed about one inch from the closed end of the combustion tube, and the other two or three inches farther up. The oxide of copper having been intro-

Fig. 34.



duced, the chloride of calcium tube and the potash apparatus are connected with the combustion tube in the usual manner. (*Fig. 24.*)

When the oxide of copper in the anterior portion of the tube is brought to a red heat, a piece of glowing charcoal is held near the bulb containing the liquid, to expel a portion of the latter and convert it into vapour. The heat applied to the bulb is gradually increased during the whole combustion; but this must be done with great caution to prevent a sudden rush of vapour, which would in all probability render the analysis worthless. It is advisable to keep a few pieces of red-hot charcoal near the closed point of the com-

bustion tube, that none of the liquid may distil and be condensed there. At the termination of the combustion, a current of air is sucked through the apparatus in the same manner as in the combustion of fixed substances.

Combustion by means of a mixture of oxide of copper with chlorate of potash. — According to M. Dumas, the carbon of an organic substance is never completely burned by oxide of copper alone, partly owing, he supposes, to the formation of a small quantity of carburet of copper with the reduced metal, and to the deposition here and there of particles of carbon which are not burned by the current of air sucked through at the end of the operation. By introducing into the closed end of the tube about two inches of a mixture consisting of one part of chlorate of potash with eight parts of oxide of copper, this source of error may be prevented to a great extent. Pure oxygen thus comes to be disengaged at the end, in which the carbon burns perfectly.

In this operation, as well as when a substance which contains a considerable proportion of nitrogen is analyzed by the ordinary method, the gas which passes through the potash apparatus becomes there saturated with moisture, which it carries away, and thus causes a deficiency in the amount of carbon. To avoid this error, a small tube, open at both ends, containing fragments of sticks of potash, is annexed to the farther extremity of the potash apparatus. The potash in that tube assumes all the water taken up by the gas, together with any trace of carbonic acid which might possibly escape the potash ley. This tube and the potash apparatus are weighed together both before and after the combustion.

Combustion by means of chromate of lead. — From the completeness with which organic bodies are burned by means of chromate of lead, that material, there is reason to believe, will supersede the use of oxide of copper to a great extent. It is obtained by precipitating the acetate or nitrate of lead by bichromate of potash, and washing the precipitate carefully with distilled water. When dry, the chromate must be

strongly calcined till it begins to melt, and be afterwards reduced to a very fine powder.

One great superiority which chromate of lead possesses over oxide of copper is in the absence of all hygrometric property; and the trace of water absorbed by the substance, while being mixed with the chromate, is very easily removed by the exhaustion.

When chromate of lead is employed, the combustion tube need not be longer than eight inches. A moderate heat is sufficient until towards the end of the process, but the heat should then be increased considerably, so as to fuse the chromate, and thus evolve pure oxygen. The increased heat renders it necessary to protect the tube by wrapping around it a thin sheet of copper, kept in its place by a few rings of iron wire. A small tube, containing fragments of sticks of potash, should be annexed to the potash apparatus to retain the moisture carried away by the gas as the latter passes through the ley.

DETERMINATION OF NITROGEN.

The nitrogen contained in organic bodies may be estimated in three different ways: first, by collecting the whole amount in the free state and measuring its volume; second, by determining the relation which exists between the nitrogen and carbonic acid gases produced by the combustion, having made a previous estimation of the carbon; and third, by converting the whole nitrogen into ammonia, which is weighed as the ammonio-chloride of platinum, or that compound is converted into metallic platinum, which is weighed.

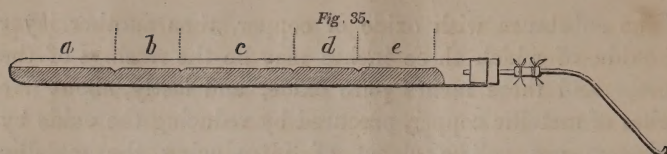
It is unnecessary to state more than the outlines of the first two of these processes as they are now likely to be superseded by the third.

When the whole nitrogen is collected in the free state, a combustion tube about twenty-four inches in length is used, five or six inches of which from the closed end are filled with carbonate of copper; above this are placed two inches of pure oxide of copper, next about six inches of the mixture

of the substance with oxide of copper, then another layer of oxide, of which three inches may be the rinsings of the mortar and three inches pure oxide, and lastly, about five inches of metallic copper, procured by reducing the oxide by hydrogen gas. The object of introducing the metallic copper is to decompose any compound of nitrogen and oxygen which may be produced during the combustion. To get rid of the air in the tube, the latter is alternately exhausted of its air by an air-pump, and filled with carbonic acid by applying heat to one half of the carbonate of copper. These operations are continued until all the air is withdrawn, and the tube is filled with pure carbonic acid gas, which is ascertained by collecting a little of the gas at the mercurial trough in a jar which contains a solution of caustic potash: if the absorption of the gas by the alkali is complete, all the air has been withdrawn.

The metallic copper and oxide in the anterior portion are first heated, and afterwards the mixture, the evolved gases being collected in a graduated mercurial jar half full of a strong solution of caustic potash. When the substance is completely burned, the remaining half of the carbonate of copper is heated to evolve carbonic acid gas, and thus drive all the nitrogen which remains in the tube forward into the receiver. The volume of the gas not absorbed by the potash, which is pure nitrogen, is then observed, and its weight calculated, the necessary corrections being made for temperature and pressure.

In the second method adverted to, the nitrogen is determined by observing the relation which exists between the carbonic acid and nitrogen gases produced by the combustion. This method is applicable to those compounds only in which the nitrogen is contained in a larger proportion than one equivalent to nine equivalents of carbon. Commonly, the evolved gas is collected at different stages of the combustion in several small graduated tubes, but it is more convenient and accurate to collect the whole gas in a single jar, which may be done in the following manner: — The first four inches (*a*, *fig.* 35.) of a combustion tube, twenty inches in length,

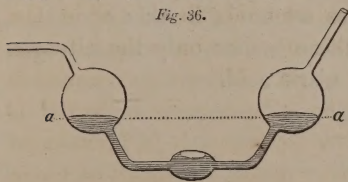


is filled with a mixture of oxide of copper with one half of the substance to be analyzed, of which about seven grains in all may be used. Next to this is placed three inches of pure oxide of copper *b*; then five inches of the mixture of oxide of copper with the remainder of the substance *c*; again, three inches of pure oxide *d*; and lastly, four inches of metallic copper *e* obtained by reduction from the oxide by hydrogen. Heat is first applied to the metallic copper and the adjoining oxide, and afterwards to the oxide in *b*. When these portions are heated to redness, heat is applied to the mixture in *a*, beginning at the closed end and proceeding gradually upwards until the portion of the substance there is completely burned, the gases which are formed being allowed to escape. By this means, all the air of the tube is expelled. The mixture in *c* is then heated by portions of half an inch at a time, commencing at the part nearest the closed end of the tube, all the gases which are produced being collected in a graduated jar at the mercurial trough. When the combustion is terminated, the volume of the mixed gases collected is observed; a strong solution of caustic potash is introduced into the jar, by means of a bent pipette, to absorb carbonic acid, and the volume of the residuary nitrogen is noticed. The atoms of nitrogen and carbon in the substance analyzed are in the same relation to each other as the volumes of the nitrogen and carbonic acid gases; hence, having determined the weight of the carbon by a previous experiment, the weight of the nitrogen may be obtained by calculation.

The third method, that in which the nitrogen is estimated by converting it into ammonia, is applicable to all azotized organic substances, excepting those in which the nitrogen exists in the form of nitric acid. The material employed is either a mixture of one part of hydrate of soda and two of lime, or one part of hydrate of potash and three

of lime. The mixture is conveniently prepared by adding the proper quantity of lime to a concentrated solution of the alkali, the strength of which has been ascertained by an alkalimetical experiment, and evaporating the mixture to dryness; or it may be made by mixing the two materials in powder intimately in a warm mortar. To have a measure of the quantity of alkaline mixture necessary, it is introduced into the combustion tube, so as to fill the latter to about three or four inches of its open end; the mixture with the weighed substance is then made in a warm and dry porcelain mortar, and introduced into the tube. From five to eight grains of the substance may be operated on. The mortar is rinsed out with an additional quantity of the mixture of alkali and lime, and the rinsings are added to the mixture in the tube, placing above the whole (when the potash mixture is used), a little asbestos to prevent the projection of any particles of the alkali. If the substance is a volatile liquid, it may be contained in small bulbs similar to those employed in the combustion of such bodies by oxide of copper. (*Fig. 32.* p. 261.) Attached to the combustion tube by

Fig. 36.



a perforated cork is a three-bulbed apparatus, represented in *fig. 36.* of the scale of a quarter of an inch to an inch, and which is filled up to *a* with pure hydrochloric

acid of specific gravity, 1.130. To ascertain whether the connection of this apparatus with the combustion tube is airtight, the heat of the hand is applied to the bulb next the tube: if the liquid rises in the opposite bulb, and does not return to its former level so long as the heat of the hand is applied, the connection is perfect. Heat is then applied to successive portions of the tube, commencing at the anterior extremity. When the whole length of the tube has been heated, air is sucked through the apparatus as in the ordinary combustion by oxide of copper, the hydrochloric acid in the bulbs is poured out into a porcelain bason, and the bulbs are washed, first with a mixture of alcohol and

ether, and lastly, with pure water. From one ounce to one ounce and a half of liquid may be used for that purpose. To the mixture of hydrochloric acid with the washings of the bulbs, a solution of pure chloride of platinum is added in excess, and the whole is evaporated to dryness. The dry residue of evaporation is treated with a mixture of two volumes of strong alcohol and one volume of ether: if this affords a yellow solution, an excess of chloride of platinum has been added, and the ammonio-chloride of platinum may be collected on a weighed filter, dried and weighed. To controul the weighing, the ammonio-chloride is calcined, in order to convert it into metallic platinum, which is also weighed: 177 parts of nitrogen are indicated by 2,788 parts of the ammonio-chloride, and by 1,233 of metallic platinum.

To insure the absence of any ammonio-chloride of platinum in the chloride employed in the operation, the solution of the chloride should be evaporated to dryness, and the residue re-dissolved in a mixture of alcohol and ether. MM. Will and Varrentrap, to whom we are indebted for this valuable process, state that substances containing cyanogen evolve all their nitrogen as ammonia in this operation, and that it is inapplicable to those bodies only the nitrogen of which exists in the form of nitric acid.

APPENDIX.

METHOD OF CALCULATING THE ATOMIC CONSTITUTION OF A BODY FROM ITS PER-CENTAGE COMPOSITION.

THE result of a quantitative analysis is first stated in the composition of 100 parts of the body analyzed. If the compound possesses a definite composition, that is, if it is not a mere mechanical mixture of its constituents, there remains to be discovered the *atomic constitution* of the substance, or *the number of equivalents of each constituent contained in one equivalent of the compound*. The equivalent of a compound body is the sum of the equivalents of its constituents; if, therefore, the equivalent of a compound be 100, the per-centage amount of any one of the constituents must either be the single equivalent number of that constituent, or else a simple multiple of the equivalent; and consequently, when the per centage is divided by the equivalent it should give the number of atoms of the particular constituent contained in one atom (100) of the compound. In like manner, if the equivalent of a compound be more or less than 100, the per-centage amounts of the ingredients, when divided by the respective single equivalent numbers, give the proportions in which the atoms exist with relation to each other, instead of the actual number of atoms, which last is then obtained by a simple calculation. This will be more obvious by an illustration. The analysis of 20 grains of crystallized sulphate of nickel gives:—

Oxide of nickel	-	-	5.34 grains
Sulphuric acid	-	-	5.70 „
Water	-	-	8.96 „
			20.00 „

Or in 100 parts, —

Oxide of nickel	-	-	26.70 „
Sulphuric acid	-	-	28.50 „
Water	-	-	44.80 „
			100.00 „

Now the equivalent of oxide of nickel is 469.68; the equivalent of sulphuric acid is 501.16; and the equivalent of water is 112.48. Then the per-centage of oxide of nickel, 26.70, divided by the equivalent of oxide of nickel, 469.68, gives the number .0568; the per-centage of sulphuric acid, 28.50, divided by the equivalent of sulphuric acid, 501.16, gives the number .0568; and the per-centage of water, 44.80, when divided by the equivalent of water, 112.48, gives the number .3982.

The proportions, therefore, of the atoms are :—

.0568 of oxide of nickel,
 .0568 of sulphuric acid, and
 .3982 of water.

Numbers which correspond to —

1 atom of oxide of nickel,
 1 atom of sulphuric acid,
 7 atoms of water.

An analysis of sugar gave as the composition of that substance in 100 parts :—

Carbon	-	-	-	42.4
Hydrogen	-	-	-	6.5
Oxygen	-	-	-	51.1
				100.0;

which quantities of carbon, hydrogen, and oxygen, when divided by the equivalents of these elements, give :—

.558 atoms of carbon,
 .520 atoms of hydrogen, and
 .511 atoms of oxygen.

The nearest corresponding whole numbers (allowing for the usual slight excess of hydrogen) being 12 of carbon, 11 of hydrogen, and 11 of oxygen.

METHODS OF TAKING THE SPECIFIC GRAVITY OF BODIES.

By the specific gravity or density of a substance is understood the relation which exists between its volume and its weight. If one body is found to possess twice the weight of another body, the bulks of the two being equal, one is said to have twice the density or specific gravity of the other, and thus in proportion; so that the volumes being equal, the densities of

bodies are directly as their weights; or the weights being equal, the densities are inversely as the volumes. For convenience in comparison, *the densities of solids and liquids are referred to pure water, and the densities of gases to dry air, as the standards of unity*; hence the number representing the specific gravity of a solid or liquid is the number of times it is heavier than water, and the specific gravity of a gas or a vapour is the number of times it is heavier than air, both being taken at the temperature of 60° Fahr., and under a pressure of 30 inches of the barometer.

In taking the density of a body, the experiment is to ascertain either the weight of a given volume or the volume of a given weight, and then to compare the weight with that of an equal bulk of the standard; that is, either of water or air.

1. *Liquids*. — To take the specific gravity of a liquid, it is brought to the temperature of 60° Fahr., and introduced into a counterpoised phial or flask, which is capable of holding, when quite full, exactly 1000 grains of distilled water at the temperature of 60° Fahr. The bottle being counterpoised, the liquid is then weighed. As the bottle holds 1000 grains of water, the weight of the liquid gives its specific gravity. Thus if the liquid operated on is proof spirit, it would be found to weigh very nearly 918·6 grains; if oil of vitriol, it would weigh 1845 grains; and if mercury, it would weigh 13,500 grains, which numbers are the densities of these liquids, water being taken as 1000; or if water is taken as 1, the decimal point is advanced three figures to the left.

Notwithstanding the simplicity of the above method, it is not generally followed, from the inconveniently large size of a bottle holding so much as 1000 grains. A much smaller bottle is preferred, weighing from 100 to 200 grains, and holding, when full, from 200 to 300 grains of water. It should be provided with a stopper formed from a piece of thermometer tube, the bore in which allows the excess of liquid to exude when the stopper is applied to the filled bottle. The weight of the flask in a perfectly clean and dry state, and also its weight when full of distilled water at 60° having been observed, the weight of water it holds is ascertained. Then, by deducting the weight of the bottle from its weight when full of the liquid whose density is to be taken, the weight of the latter is obtained, and its density may be calculated, by the rule, — As the weight of water is to the weight of liquid, so 1 is to the density of the liquid. That is, the density of the liquid is obtained by dividing its weight by the weight of the water.

2. *Solids heavier than water*. — The solid whose density is re-

quired is first weighed in air, and afterwards (if insoluble) in distilled water at 60° , being suspended from the pan of the balance by a fibre of unspun silk. The weight of the substance in water is found to be less than in air; and the weight lost by immersion represents the weight of the bulk of water which the body displaces, according to the hydrostatic law, that the weight of a substance in any medium (such as water in the present instance) is less than its absolute weight by the weight of the bulk of the medium which it displaces; and obviously, it must displace its own bulk. We thus obtain the two data necessary to calculate the density of a solid; namely, the weight of the substance in air, and also the weight of its own bulk of water. Suppose a substance which weighs 420 grains in air to weigh 345 grains in water; then the weight lost by immersion being 75 grains, we have the proportion, —

$$\text{as } 75 : 420 :: 1 : 5.6;$$

5.6 is therefore the density of the substance in question.

Another method of obtaining the specific gravity of an insoluble substance is by the use of the bottle employed in taking the specific gravity of liquids. A known weight of the substance is introduced into the bottle, which is completely filled with water and weighed. The weight of the bottle when containing both the substance and water is subtracted from the sum of the weights of the substance in air and the bottle when containing water only; the remainder is the weight of the water which the solid displaces. For example, suppose the weight of the substance in air to be 180 grains, the weight of the bottle when filled with water 240 grains, and the weight of the bottle when containing both the substance and water 390 grains: then from 420 ($180 + 240$) subtract 390, the remainder, 30, is the weight of the volume of water displaced by 180 grains of the substance; and hence the proportion as $30 : 1 :: 180 : 6$. The specific gravity required is therefore 6.

If the solid whose density is to be taken is soluble in water, some other fluid of known specific gravity must be used in which it is insoluble. Either oil of turpentine, alcohol, or olive oil will generally be found available. The specific gravity of the substance is then found by the following proportion, — As the density of water is to the density of the liquid used, so is the density of the substance in relation to the liquid in which it is weighed as unity, to its density compared with water as unity.

If, for example, a body is found to possess the density 3, compared with olive oil as unity, then olive oil being .91 compared with water as 1, we have the proportion, as 1 (the assumed density of water) is to .91, so is 3 to 2.73, which is the specific gravity required.

3. *Solids lighter than water.*—When the solid whose density is required is lighter than water, it should be attached by means of a slender thread to a piece of a heavier solid of sufficient weight that both when together shall sink in water. The points to be determined experimentally in such a case are;—

- 1°. The weight of the light solid in air;
- 2°. The weight of the heavy solid in air;
- 3°. The weight of the heavy solid in water; and
- 4°. The weight of the two together in water.

The difference between the weight of both bodies tied together in water, and the sum of their weights in air, shows the weight of the whole bulk of water displaced by both. To know how much of this is due to the heavy body, the weight of the latter in water is subtracted from its weight in air, the remainder is the weight of the water displaced by the heavy body alone, which, deducted from the entire weight of water displaced by both, leaves that displaced by the light body alone. The rule, therefore, is—from the difference between the weight of the two in water and their weight in air, subtract the difference between the weight of the heavy solid in air and its weight in water; the remainder is the weight of a quantity of water equal in bulk to the light solid, from which the specific gravity of the substance may be obtained by simple proportion. As an example, suppose the following case,—

- | | | | |
|---|---|---|-----------|
| 1°. The weight of the light solid in air | - | - | 12 grains |
| 2°. The weight of the heavy solid in air | - | - | 22 „ |
| 3°. The weight of the heavy solid in water | - | - | 19 „ |
| 4°. The weight of both tied together in water | - | - | 8 „ |

Then, from the weight of both in air (12+22) - 34 grains

Deduct the weight of both in water	-	-	8 „
			— „
			26 „

And from the remainder deduct 22-19=3 - 3 „

Which gives the weight of the bulk of water	}	-	23 „
displaced by the light body alone	-	-	

The following proportion then affords the specific gravity of the body:—

$$\text{as } 23 : 12 :: 1 : 0.5217.$$

4. *Gases.*—The standard of comparison to which the densities of gases and vapours are referred is air, as it would be inconvenient from the extreme lightness of these bodies to compare their weights with that of an equal bulk of water. From the careful experiments of Dr. Prout it appears that 100 cubic inches of atmospheric air deprived of carbonic acid and aqueous vapour weighs 31.0117 grains at 30 inches of the barometer and at the temperature of 60° Fahr.; from which observation it is easy to calculate the absolute weight of any bulk of a gas from its specific gravity. Thus the specific gravity of chlorine is found to be 2.47; to find how much 100 cubic inches of that gas weighs at mean temperature and pressure, we make use of the proportion,

$$\text{as } 1 : 2.47 :: 31.01 : 76.59;$$

therefore 100 cubic inches of chlorine weigh 76.59 grains.

The simplest method of obtaining the specific gravity of a gas is the following:—The object is to ascertain the weight of a bulk of gas equal to the bulk of a known weight of air. For this purpose, a light glass globe furnished with a stop-cock, is very accurately weighed when full of air, then exhausted of its air by connecting it with an air-pump, and weighed in the vacuous state. The weight of the air withdrawn by the exhaustion is thus ascertained. The globe, still vacuous, is connected with a jar containing the gas which is to be weighed, at the water or mercurial trough; the jar having a stop-cock at its top into which the stop-cock of the globe can be screwed air-tight. On gently opening both stopcocks, a quantity of gas rushes from the jar into the exhausted globe, equal in bulk to the air withdrawn by the exhaustion, if the surface of the liquid within the jar be brought to the level of that without in the trough, and the temperature of the air and the barometric pressure have not varied during the experiment. The stopcock being closed, the globe is detached from the jar, and weighed. The difference between its weight when containing the gas and when vacuous is the weight of a bulk of the gas equal to the bulk of air whose place it occupies, the weight of which has already been determined.

Suppose the globe to lose 10.33 grains by exhaustion of air, and when exhausted, to gain 15.78 grains by admitting carbonic acid

gas; then, assuming 1· as the density of air, we have the proportion,

$$\text{as } 10\cdot33 : 15\cdot78 :: 1 : 1\cdot527;$$

the specific gravity of carbonic acid gas is, therefore, 1·527.

Although thus simple in principle, the operation in its details is one of extreme delicacy. From the facility with which gases undergo a change in their bulk through variations of temperature and pressure, it is obvious that if the temperature and barometric pressure vary during the course of the experiment, corrections must be made. As an illustration of the necessary corrections, suppose the bulk of air to weigh 12 grains at the temperature of 60° Fahr., and under a pressure of 30 inches bar.; and the same bulk of the gas whose density is required to weigh 20 grains, but at the temperature of 50° Fahr. and under a pressure of 28 inches bar. The points to be determined here are two:—

1°. Considering the volume of the air withdrawn and the gas admitted as 1; at the observed temperatures and pressures, what would be the volume of the gas at the temperature and pressure at which the air was weighed?

And, 2°, having obtained that volume, what is the corresponding increase or reduction in the *weight* of the gas?

Performed according to rules which are given in the note below*, the results of these calculations are as follow:—

* 1. *For changes in bulk by pressure.* The volume which a gas should possess at one pressure may be calculated from its known volume at another pressure by the use of the following proportion:—As the pressure to which the gas is to be corrected is to the observed pressure, so is the observed volume to the volume required. In the example in the text (*b*), the pressure to which the gas is to be reduced is 30 inches, the observed pressure 28 inches, and the volume 1·019. Then as $30 : 28 :: 1\cdot019 : 0\cdot951$.

2. *For changes in bulk by temperature.* From the very recent experiments of M. Regnault, it appears that a volume of gas expands by heat $\frac{1}{459}$ of its bulk for each degree Fahrenheit. Hence, the volume of a gas at 0° Fahr. being 1, at any higher temperature it is found by the formula $1 + \frac{\text{Temp. Fahr.}}{459}$. The determination of the volume of a gas at one temperature from its known volume at another temperature may be attained by the following formula:—Let t be the temperature Fahrenheit at which the volume of the gas is observed; v the temperature Fahrenheit to which the volume of the gas is to be reduced; x the observed volume at t ; and y the volume at v required;

$$\text{Then } y = \frac{(459 + v) \times x}{459 + t}$$

3. It is frequently necessary to combine corrections both for temperature and pressure. In such a case, as in the example in the text, the reduction of volume is first made for temperature, and that corrected volume is afterwards reduced according to the pressure.

(a) A volume of gas equal to 1· at 50° Fahr. is equal to 1·019 at 60° Fahr.

(b) A volume of gas equal to 1·019 at 28 inches of the barometer is equal to 0·951 at 30 inches.

A volume of the gas, therefore, equal to 0·951 weighs 20 grains, a volume of air equal to 1· at the same temperature and pressure weighing 12 grains. Then, if 0·951 vol. weighs 20 grains, 1 vol. should weigh 21·03 grains: and

$$\text{as } 12 : 1 :: 21\cdot03 : 1\cdot75;$$

1·75 is therefore the density required.

The state of dryness of a gas is another circumstance which interferes with its volume; for which reason, due care should be taken to insure either the perfect dryness of the gas, or its complete saturation with moisture. In the latter case the temperature must be noticed and the observed volume reduced according to the proportion of aqueous vapour capable of existing in the gas at the observed temperature. The proportions of vapour by volume contained in 1 vol. of the saturated gas for temperatures between 40° and 80° Fahr. are expressed in the following table* (*Faraday's Manipulation*).

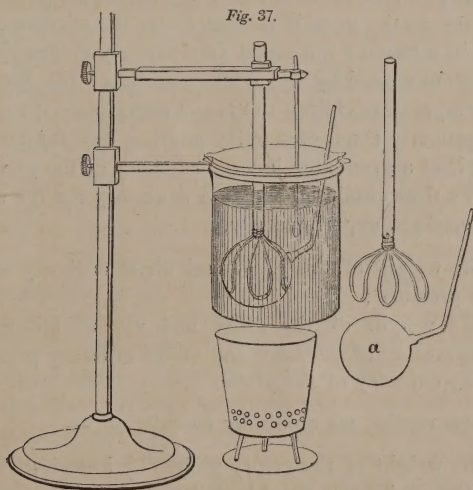
40°	·00933	54°	·01533	68°	·02406
41	·00973	55	·01586	69	·02483
42	·01013	56	·01640	70	·02566
43	·01053	57	·01693	71	·02653
44	·01093	58	·01753	72	·02740
45	·01133	59	·01810	73	·02830
46	·01173	60	·01866	74	·02923
47	·01213	61	·01923	75	·03020
48	·01253	62	·01980	76	·03120
49	·01293	63	·02050	77	·03220
50	·01333	64	·02120	78	·03323
51	·01380	65	·02190	79	·03423
52	·01426	66	·02260	80	·03533
53	·01480	67	·02330		

The preceding method of obtaining the density of a gas still requires a slight correction from another circumstance when the temperature and pressure differ considerably at the time of weighing the air and at the time of weighing the gas, but one so trifling that it may, in general, be neglected. The necessity of this correction arises from the impossibility of obtaining a perfect vacuum in the globe; and the remaining small quantity of air may

* A cubic inch of aqueous vapour corrected to the temperature of 60°, and at a pressure of 30 inches, weighs 0·1929 grains.

occupy a different space when weighed with the gas to that which it occupied when the globe was weighed with air, and consequently, the bulk of the gas admitted into the globe is not the same as the bulk of the air withdrawn. If the amount of rarefaction of the air in the exhausted flask is observed by means of a barometer gauge attached to the air-pump, the amount of the remaining air may be calculated when the weight of the quantity withdrawn is ascertained; then the alteration to which it would be subject in bulk by changes of temperature and pressure may also be estimated and a due allowance made on the bulk of the gas admitted into the globe.

5. *Method of taking the density of vapours.*— Few processes of recent invention have been of more assistance in the prosecution of chemical research than the method contrived by M. Dumas for obtaining the density of the vapour of a volatile body. Neglecting the minute, but in general unnecessary, precautions requisite to insure results of mathematical accuracy, the process is briefly the following:— A light glass globe (*a*, *fig. 37.*), having a capacity of



from twelve to sixteen fluid ounces, is drawn out at its neck to a narrow tube six or eight inches long, as shown in the figure; the point of the tube is cut across with a file and fused but not sealed. The globe is then weighed, containing, as usual, atmospheric air, and the temperature and pressure at the time are observed. To

introduce a volatile liquid into the globe, the latter is warmed so as to expel a portion of its air, and the end of the beak is dipped under the surface of the liquid. As the globe cools, the air within contracts, and the liquid is sucked up. When a sufficient quantity of the liquid has entered (from 100 to 150 grains), the globe is enclosed in a sort of wire cage, as represented in the figure, and immersed in a bath of some material which can be heated to 50° or 60° above the boiling point of the substance. Either water, oil, chloride of calcium, chloride of zinc, or the fusible alloy of bismuth, tin, and lead may be used for this purpose. When the temperature of the bath rises above the boiling point of the substance, a stream of vapour rushes through the tube, carrying with it all the air of the globe, if sufficient quantity of the substance had been introduced. When no more vapour is emitted, the point of the tube is sealed before the flame of the blowpipe, the temperature of the bath being observed at the same moment*, and the globe is removed from the bath, washed, dried, and weighed.

The next point to be determined is the capacity of the globe. For this purpose the neck is broken under the surface of mercury contained in a bason; the metal immediately enters the globe to fill the vacuum caused by the condensation of the vapour at the ordinary temperature, filling the vessel completely if the operation has been properly conducted. By pouring out the mercury, and measuring it in a graduated jar, the capacity of the globe is ascertained. We thus obtain all the data necessary for the calculation, having determined experimentally,

- 1°. The weight of the globe and air at ordinary temperature and pressure;
- 2°. The weight of the globe and vapour filling it at the temperature of the bath and under ordinary pressure; and,
- 3°. The capacity of the globe.

Having these results, we obtain by calculation, —

- 1°. The weight of the empty globe (by knowing the capacity of the globe the weight of the air filling it can be calculated, which, deducted from the weight of the globe and air, leaves the weight of the globe when vacuous);
- 2°. The weight of vapour filling the globe at the temperature

* The globe should be considered to possess a temperature 1° less than that of the bath.

- of the bath (by deducting the weight of the empty globe from the weight of the globe and vapour); and,
- 3^o. The weight of air filling the globe at the temperature of the bath, and at the pressure at which the globe was sealed with the vapour.

The last calculation is made according to rules given in the note, page 275.; having performed which, the density of the vapour required is obtained by the simple proportion, — As the weight of air filling the globe at the temperature of the bath is to the weight of vapour filling the globe at the same temperature, so is 1 to the density required.

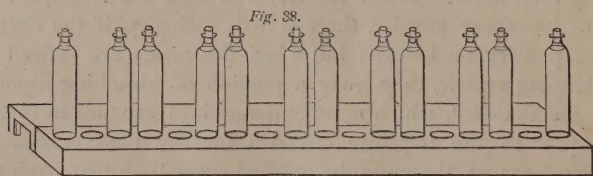
APPENDIX ON CHLORIMETRY.

The following observations by Mr. Crum describe a method of testing weak solutions of chloride of lime, such as are used in bleaching, which appears to be well adapted to the use of the practical bleacher.* “Chlorimetry requires to be practised by the bleacher for two purposes — First, he has to learn the commercial value of the bleaching powder which he purchases; and with that view he can scarcely desire any thing better than the method either by arsenious acid, or green copperas. But the more important, because the hourly testing of his bleaching liquor, and that on which the safety of his goods depends, is the ascertaining the strength of the weak solutions in which the goods have to be immersed. If the solution is too strong, the fabric is apt to be injured. If too weak, parts of the goods remain brown, and the operation must be repeated. The range within which cotton is safe in this process is not very wide. A solution standing 1° on Twaddell's hydrometer, (spec. grav. 1.005) is not more than safe for such goods, while that of half a degree is scarcely sufficient for the first operation of stout cloth, unless it is packed more loosely than usual. When the vessel is first set with fresh solution of bleaching powder, there is little difficulty, if the character of the powder be known; but when the goods are retired from the steeping vessels, they leave a portion of bleaching liquor behind, unexhausted, which must be taken into account in restoring the liquor to the requisite strength for the next parcel. The chlorimeter must, therefore, be applied every time that fresh goods are put into the liquid.

* From the proceedings of the Glasgow Philosophical Society.

" I introduced another into our establishment some years ago, which has been in regular use ever since, and by which the testing is performed in an instant. It depends on the depth of colour of the peracetate of iron. A solution is formed of proto-chloride of iron, by dissolving cast-iron turnings in muriatic acid, of half the usual strength. To ensure perfect saturation, a large excess of iron is kept for some time in contact with the solution at the heat of boiling water. One measure of this solution, at 40° Twaddell, (spec. grav. 1.200) is mixed with one of acetic acid, such as is sold at 8s. a gallon. That forms the proof solution. If mixed with six or eight parts of water it is quite colourless, but chloride of lime occasions with it the production of peracetate of iron, which has a peculiarly intense red colour.

" A set of phials is procured, 12 in number, all of the same diameter. A quantity of the proof solution, equal to $\frac{1}{12}$ th of their capacity is put into each, and then they are filled up with bleaching liquor of various strengths, the first at $\frac{1}{12}$ th of a degree of Twaddell, the second, $\frac{2}{12}$ ths, the third, $\frac{3}{12}$ ths, and so on up to $\frac{12}{12}$ ths, or 1 degree. They are then well corked up, and ranged together, two and two, in a piece of wood, in holes drilled to suit them. We have thus a series of phials, showing the shades of colour which those various solutions are capable of producing. To ascertain the strength of an unknown and partially exhausted bleaching liquor, the proof solution of iron is put into a phial similar to those in the instrument, up to a certain mark, $\frac{1}{12}$ th of the whole. The phial is then filled up with the unknown bleaching liquor, shaken, and placed beside that one in the instrument which most resembles it. The number of that phial is its strength in 12ths of a degree of the hydrometer; and, by inspecting the annexed table, we find at once how much of a solution of bleaching powder, which is always kept in stock, at a uniform strength of six degrees, is necessary to raise the whole of the liquor in the testing vessel to the desired strength.



" The instrument is formed of long 2-ounce phials cast in a mould; those of blown glass not being of uniform diameter. The

outside, which alone is rough, is polished by grinding, and in this state they can easily be procured at 4s. 6d. a dozen. They are placed two and two, so that the bottle containing the liquid to be examined may be set by the side of any one in the series, and the colour compared by looking through the liquid upon a broad piece of white paper stretched upon a board behind the instrument.

“To explain the table it is necessary to state that the steeping vessels we employ contain, at the proper height for receiving goods, 1440 gallons, or 288 measures of 5 gallons each, — a measure being the quantity easily carried at a time. In the following table, 0 represents water, and the numbers 1, 2, 3, &c. are the strength of the liquor already in the vessel in 12ths of a degree of Twaddell, as ascertained by the chlorimeter. If the vessel has to be set anew, we see by the first table that 32 measures of liquor at 6° must be added to (256 measures of) water to produce 288 measures of liquor at $\frac{8}{12}$ ths of a degree. But if the liquor already in the vessel is found by the chlorimeter to produce a colour equal to the second phial, then 24 measures only are necessary, and so on.”

To stand $\frac{8}{12}$ °				To stand $\frac{6}{12}$ °			
0 requires 32 measures.				0 requires 24 measures.			
1	—	28	—	1	—	20	—
2	—	24	—	2	—	16	—
3	—	20	—	3	—	12	—
4	—	16	—	4	—	8	—
5	—	12	—	5	—	4	—
6	—	8	—				
7	—	4	—				
To stand $\frac{4}{12}$ °				To stand $\frac{3}{12}$ °			
0 requires 16 measures.				0 requires 12 measures.			
1	—	12	—	1	—	8	—
2	—	8	—	2	—	4	—
3	—	4	—				

TABLES FOR CALCULATIONS IN ANALYSIS.

In quantitative analysis the substance whose weight is to be determined, instead of being weighed in its free or uncombined state, is more frequently weighed in a state of union with another body forming a definite compound and one of known composition, and from the weight of which the weight of the particular constituent whose amount is the object of research is calculated. To facilitate calculations of this kind the following tables have been constructed to express the quantities of one substance contained in 1·00000, 2·00000, 3·00000, 4·00000, 5·00000, 6·00000, 7·00000, 8·00000, and 9·00000 parts respectively of another substance; and by adding together the proportions corresponding to all the figures in the number which expresses the weight of a substance, the proportion in which one body is contained in or indicated by any amount of another body may be ascertained. The first column, headed "Found," contains the names of the substances weighed; the second, headed "Required," contains the names of the substances whose amounts are to be determined; and the remaining columns, headed by the first nine whole numbers, express the proportions of the "required" substance contained in or indicated by as many parts of the "found" substance as the number at the head of the column. When the number which expresses the quantity of the substance found contains more than one figure as its whole number, the decimal point is advanced in proportion to the right; but if less than one whole number, the decimal point is advanced to the left. The application of these tables will be better comprehended by an example. To find the amount of chlorine contained in 21·34 grains of chloride of silver proceed as follows:—

20·00	The number in the column 2, with the decimal point advanced one figure to the right	-	4·9340
1·	The number in column 1, without changing the decimal point	-	·2467
·3	The number in column 3, with the decimal point advanced one figure to the left	-	·0740
·04	The number in column 4, with the decimal point advanced two figures to the left	-	·0098
21·34	grains of chloride of silver contain grains of chlorine	-	5·2645

Found.	Required.	1.	2.	3.	4.	5.	6.	7.	8.	9.
Alumina. Al_2O_3 .	Aluminum. Al_2 .	0.53295	1.06591	1.59886	2.13181	2.66476	3.19772	3.73067	4.26362	4.79658
Antimony, ox. of. Sb O_5 .	Antimony. Sb .	0.84317	1.68634	2.52951	3.37268	4.21585	5.05902	5.90219	6.74536	7.58853
Antimony, sulphuret of. Sb S_3 .	Antimony. Sb .	0.72771	1.45542	2.18313	2.91084	3.63855	4.36626	5.09397	5.82168	6.54939
Antimony, sulphuret of. Sb S_3 .	Ox. of antimony. Sb O_3 .	0.86307	1.72614	2.58921	3.45228	4.31535	5.17842	6.04149	6.90456	7.76763
Antimony. Sb .	Ox. of antimony. Sb O_3 .	1.18600	2.37200	3.55800	4.74400	5.93000	7.11600	8.30200	9.48800	10.67400
Arsenic, sulphuret of. As S_3 .	Arsenic. As .	0.60903	1.21806	1.82709	2.43612	3.04515	3.65418	4.26321	4.87224	5.48127
Arsenic, sulphuret of. As S_3 .	Arsenious acid. As O_3 .	0.80338	1.60676	2.41014	3.21352	4.01690	4.82028	5.62366	6.42704	7.23042
Arsenic, pentasulphuret of. As S_5 .	Arsenic. As .	0.48311	0.96622	1.44933	1.93244	2.41555	2.89866	3.38177	3.86488	4.34799
Arsenic, pentasulphuret of. As S_5 .	Arsenic acid. As O_5 .	0.74006	1.48012	2.22018	2.96024	3.70030	4.44036	5.18042	5.92048	6.66054

Found.	Required.	1.	2.	3.	4.	5.	6.	7.	8.	9.
Barytes. Ba O.	Barium. Ba.	0·89549	1·79099	2·68648	3·58198	4·47747	5·37296	6·26846	7·16395	8·05945
Barytes, sulphate of. Ba O, S O ₃ .	Barytes. Ba O.	0·65628	1·31256	1·96884	2·62512	3·28140	3·93768	4·59396	5·25024	5·90652
Barytes, car- bonate of. Ba O, C O ₂ .	Barytes. Ba O.	0·77586	1·55172	2·32758	3·10344	3·87930	4·65516	5·43102	6·20688	6·98274
Barium, silico- fluoride of. Ba ₃ F ₃ + Si ₂ F ₆ .	Barytes. 3 Ba O.	0·18297	0·36594	0·54891	0·73188	0·91485	1·09782	1·28079	1·46376	1·64673
Bismuth, oxide of. Bi O.	Bismuth. Bi.	0·89867	1·79735	2·69602	3·59470	4·49337	5·39204	6·29072	7·18939	8·08807
Bromide of silver. Ag Br.	Bromine. Br.	0·41989	0·83978	1·25967	1·67956	2·09945	2·51934	2·93923	3·35912	3·77901
Cadmium, oxide of. Cd O.	Cadmium. Cd.	0·87449	1·74899	2·62348	3·49797	4·37246	5·24696	6·12145	6·99594	7·87044
Carbonic acid. C O ₂ .	Carbon. C.	0·27272	0·54544	0·81816	1·09088	1·36360	1·63632	1·90904	2·18176	2·45448
Carbonic acid. C O ₂ .	Oxalic acid. $\frac{1}{2}(\text{C}_2\text{O}_5)$	0·81818	1·63636	2·45454	3·27272	4·09090	4·90908	5·72726	6·54544	7·36362

Found.	Required.	1.	2.	3.	4.	5.	6.	7.	8.	9.
Carbonate of lime. Ca O, C O ₂ .	Oxalic acid. $\frac{1}{2}$ (C ₂ O ₃).	0·35656	0·71312	1·06968	1·42624	1·78280	2·13936	2·49592	2·85248	3·20904
Carbonate of lime. Ca O, C O ₂ .	Carbonic acid. C O ₂ .	0·43579	0·87158	1·30737	1·74316	2·17895	2·61474	3·05053	3·48632	3·92211
Carbonate of barytes. Ba O, C O ₂ .	Carbonic acid. C O ₂ .	0·22323	0·44646	0·66969	0·89292	1·11615	1·33938	1·56261	1·78584	2·00907
Chloride (sub) of mercury. Hg ₂ Cl.	Chlorine. Cl.	0·14883	0·29766	0·44649	0·59532	0·74415	0·89298	1·04181	1·19064	1·33947
Chloride of potassium. K Cl.	Chlorine. Cl.	0·47466	0·94932	1·42398	1·89864	2·37330	2·84796	3·32262	3·79728	4·27194
Chloride of silver. Ag Cl.	Chlorine. Cl.	0·24670	0·49340	0·74010	0·98680	1·23350	1·48020	1·72690	1·97360	2·22030
Chloride of silver. Ag Cl.	Hydrochloric acid. H Cl.	0·25366	0·50732	0·76098	1·01464	1·26830	1·52196	1·77565	2·02928	2·28294
Chloride of sodium. Na Cl.	Chlorine. Cl.	0·60344	1·20688	1·81032	2·41376	3·01720	3·62064	4·22408	4·82752	5·43096
Chromium, oxide of. Cr ₂ O ₃ .	Chromium. Cr ₂ .	0·75109	1·40217	2·10326	2·80435	3·50543	4·20652	4·90761	5·608 0	6·30978

Found.	Required.	1.	2.	3.	4.	5.	6.	7.	8.	9.
Chromium, oxide of. Cr_2O_3 .	Chromic acid. $2(\text{Cr O}_3)$.	1.29891	2.59782	3.89673	5.19564	6.49455	7.79346	9.09237	10.39128	11.69019
Chromate of barytes. Ba O, Cr O_3 .	Chromic acid. Cr O_3 .	0.40518	0.81036	1.21554	1.62072	2.02590	2.43108	2.83626	3.24144	3.64662
Chromate of lead. Pb O, Cr O_3 .	Chromic acid. Cr O_3 .	0.31853	0.63706	0.95559	1.27412	1.59265	1.91118	2.22971	2.54824	2.86677
Cobalt, oxide of. Co O .	Cobalt. Co .	0.78678	1.57355	2.36033	3.14710	3.93388	4.72066	5.50743	6.29421	7.08098
Copper, protoxide of. Cu O .	Copper. Cu .	0.79826	1.59653	2.39479	3.19305	3.99131	4.78958	5.58784	6.38610	7.18437
Copper, protoxide of. Cu O .	Suboxide of copper. $\frac{1}{2}(\text{Cu}_2\text{O})$.	0.89913	1.79826	2.69739	3.59652	4.49565	5.39478	6.29391	7.19304	8.09217
Cyanide of silver. $\text{Ag, C}_2\text{N}$.	Cyanogen. C_2N .	0.19620	0.39240	0.58860	0.78480	0.98100	1.17720	1.37340	1.56960	1.76580
Cyanide of silver. $\text{Ag, C}_2\text{N}$.	Hydrocyanic acid. $\text{H, C}_2\text{N}$.	0.20362	0.40724	0.61086	0.81448	1.01810	1.22172	1.42534	1.62896	1.83258
Cyanogen. C_2N .	Carbon. C .	0.46338	0.92676	1.39014	1.85352	2.31690	2.78028	3.24366	3.70704	4.17042

Found.	Required.	1.	2.	3.	4.	5.	6.	7.	8.	9.
Cyanogen. $C_2 N_2$.	Nitrogen. N.	0·53662	1·07324	1·60986	2·14648	2·86310	3·21972	3·75634	4·29296	4·82958
Fluoride of calcium. Ca F.	Fluorine. F.	0·47732	0·95464	1·43196	1·91928	2·38660	2·86392	3·34124	3·81856	4·29588
Gold, oxide of. $Au_2 O_3$.	Gold. Au_2 .	0·89232	1·78464	2·67696	3·56928	4·46160	5·35392	6·24624	7·13856	8·03088
Gold. Au_2 .	Oxide of gold. $Au_2 O_5$.	1·12067	2·24134	3·36201	4·48268	5·60335	6·72402	7·84469	8·96536	10·08603
Hydrobromic acid. H Br.	Bromine. Br.	0·98740	1·97480	2·96220	3·94960	4·93700	5·92440	6·91180	7·89920	8·88660
Hydrochloric acid. H Cl.	Chlorine. Cl.	0·97258	1·94516	2·91774	3·89032	4·86290	5·83548	6·80806	7·78064	8·75322
Hydriodic acid. H I.	Iodine. I.	0·99216	1·98432	2·97648	3·96864	4·96080	5·95296	6·94512	7·93728	8·92944
Iodide of silver. Ag I.	Iodine. I.	0·53868	1·07736	1·61604	2·15472	2·69340	3·23208	3·77076	4·30944	4·84812
Iodide (sub) of copper. $Cu_2 I$.	Iodine. I.	0·66604	1·33208	1·99812	2·66416	3·33020	3·99624	4·66228	5·32882	5·99436

Found.	Required.	1.	2.	3.	4.	5.	6.	7.	8.	9.
Iron, protoxide of. Fe O.	Iron. Fe.	0·77232	1·54464	2·31696	3·08928	3·86160	4·63392	5·40624	6·17856	6·95088
Iron, peroxide of. Fe ₂ O ₃ .	Iron. Fe ₂ .	0·69338	1·38677	2·08015	2·77354	3·46692	4·16031	4·85369	5·54708	6·24046
Iron, peroxide of. Fe ₂ O ₅ .	Iron, protoxide of. 2 Fe O.	0·89780	1·79560	2·69340	3·59120	4·48900	5·38680	6·28460	7·18240	8·08020
Lead, oxide of. Pb O.	Lead. Pb.	0·92829	1·85658	2·78487	3·71316	4·64145	5·56974	6·49803	7·42632	8·35461
Lead. Pb.	Oxide of lead. Pb O.	1·07725	2·15450	3·23174	4·30899	5·38623	6·46348	7·54072	8·61797	9·69521
Lead, chloride of. Pb Cl.	Oxide of lead. Pb O.	0·80275	1·60550	2·40825	3·21100	4·01375	4·81650	5·61925	6·42200	7·22475
Lead, sulphate of. Pb O, S O ₅ .	Oxide of lead. Pb O.	0·73563	1·47126	2·20689	2·94252	3·67815	4·41378	5·14941	5·88504	6·62067
Lead, sulphate of. Pb O, S O ₅ .	Lead. Pb.	0·68287	1·36574	2·04861	2·73148	3·41435	4·09722	4·78009	5·46296	6·14583
Lime. Ca O.	Calcium. Ca.	0·71911	1·43823	2·15735	2·87646	3·59558	4·31470	5·03381	5·75293	6·47204

Found.	Required.	1.	2.	3.	4.	5.	6.	7.	8.	9.
Lime, sulphate of. Ca O, S O_3 .	Lime. Ca O .	0·41532	0·83064	1·24596	1·66128	2·07660	2·49102	2·90724	3·32256	3·73788
Lime, carbonate of. Ca O, C O_2 .	Lime. Ca O .	0·56292	1·12584	1·68876	2·25168	2·81460	3·37752	3·94044	4·50336	5·06628
Lithia, phosphate of soda and. $(2\text{Li O} + \text{P O}_5) + (2\text{Na O} + \text{P O}_5)$.	Lithia. 2 Li O .	0·12381	0·24762	0·37143	0·49524	0·61905	0·74286	0·86667	0·99048	1·11429
Magnesia. Mg O .	Magnesium. Mg .	0·61293	1·22587	1·83880	2·45173	3·06466	3·67760	4·29053	4·90346	5·51640
Magnesia, phosphate of. $2\text{Mg O} + \text{P O}_5$.	Magnesia. 2 Mg O .	0·36671	0·73342	1·10013	1·46684	1·83355	2·20026	2·56697	2·93368	3·30039
Manganese, protoxide of. Mn O .	Manganese. Mn .	0·77573	1·55147	2·32720	3·10294	3·87867	4·65440	5·43014	6·20587	6·98161
Manganic oxide. Mn_2O_3 .	Manganese. Mn_2 .	0·69752	1·39504	2·09256	2·79008	3·48760	4·18512	4·88264	5·58016	6·27768
Manganese, peroxide of. Mn O_2 .	Manganese. Mn .	0·53533	1·07106	1·60660	2·14213	2·67766	3·21319	3·74872	4·28426	4·81979
Manganomanganic oxide. $\text{Mn O} + \text{Mn}_2\text{O}_3$.	Protoxide of manganese. 3 Mn O .	0·93044	1·86088	2·79132	3·72176	4·65220	5·58264	6·51308	7·44352	8·37396

Found.	Required.	1.	2.	3.	4.	5.	6.	7.	8.	9.
Mercury. Hg ₂ .	Suboxide of mercury. Hg ₂ O.	1·03950	2·07900	3·11850	4·15800	5·19750	6·23700	7·27650	8·31600	9·35550
Mercury. Hg.	Protoxide of mercury. Hg O.	1·07900	2·15800	3·23700	4·31600	5·39500	6·47400	7·55300	8·63200	9·71100
Mercury. Hg.	Subchloride of mercury. $\frac{1}{2}$ (Hg ₂ Cl).	1·17485	2·34970	3·52455	4·69940	5·87425	7·04910	8·22395	9·39880	10·57365
Mercury. Hg.	Chloride of mercury. Hg Cl.	1·34969	2·69938	4·04907	5·39876	6·74845	8·09814	9·44783	10·79752	12·14721
Mercury, sub- oxide of. Hg ₂ O.	Mercury. Hg ₂ .	0·96200	1·92400	2·88600	3·84800	4·81000	5·77201	6·73401	7·69601	8·65801
Mercury, pro- toxide of. Hg O.	Mercury. Hg.	0·92678	1·85357	2·78035	3·70714	4·63392	5·56070	6·48749	7·41427	8·34106
Mercury, sub- chloride of. Hg ₂ Cl.	Mercury. Hg ₂ .	0·85117	1·70234	2·55351	3·40468	4·25585	5·10702	5·95819	6·80936	7·66053
Mercury, sul- phuret of. Hg S.	Mercury. Hg.	0·86287	1·72574	2·58861	3·45148	4·31435	5·17722	6·04009	6·90296	7·76583
Mercury, sul- phuret of. Hg S.	Protoxide of mercury. Hg O.	0·93104	1·86208	2·79312	3·72416	4·65520	5·58624	6·51728	7·44832	8·37936

Found.	Required.	1.	2.	3.	4.	5.	6.	7.	8.	9.
Molybdic acid. Mo O ₃ .	Molybdenum. Mo.	0·66612	1·33224	1·99836	2·66448	3·33059	3·99671	4·66283	5·32895	5·99507
Molybdenum, tersulphuret of. Mo S ₃ .	Molybdenum. Mo.	0·49793	0·99586	1·49379	1·99172	2·48965	2·98758	3·48551	3·98344	4·48137
Nickel, oxide of. Nk O.	Nickel. Nk.	0·78709	1·57417	2·36126	3·14835	3·93543	4·72252	5·50961	6·29670	7·08378
Nitrogen. N.	Ammonia. NH ₃ .	1·21147	2·42294	3·63441	4·84588	6·05735	7·26882	8·48029	9·69176	10·90323
Nitrogen. N.	Cyanogen. NC ₂ .	1·86351	3·72702	5·59053	7·45404	9·31755	11·18106	13·04457	14·90808	16·77159
Nitric acid. N O ₅ .	Nitrous acid. NO ₃ .	0·70459	1·40918	2·11377	2·81836	3·52295	4·22754	4·93213	5·63672	6·34131
Nitrate of barytes. Ba O, NO ₅ .	Nitric acid. NO ₅ .	0·41436	0·82872	1·24308	1·65744	2·07180	2·48616	2·90052	3·31488	3·72924
Sulphate of barytes. Ba O, SO ₅ .	Nitric acid. NO ₅ .	0·46434	0·92868	1·39302	1·85736	2·32170	2·78604	3·25038	3·71472	4·17906
Phosphoric acid. PO ₅ .	Phosphorus. P.	0·43966	0·87931	1·31897	1·75863	2·19828	2·63794	3·07760	3·51726	3·95691

Found.	Required.	1.	2.	3.	4.	5.	6.	7.	8.	9.
Phosphoric acid. PO_5 .	Hypophosphorous acid. PO_5 .	0.55173	1.10346	1.65519	2.20692	2.75865	3.31038	3.86211	4.41384	4.96557
Phosphoric acid. PO_5 .	Phosphorous acid. PO_3 .	0.77586	1.55172	2.32758	3.10344	3.87930	4.65516	5.43102	6.20688	6.98274
Phosphate of barytes. $2 \text{Ba O} + \text{PO}_3$.	Phosphoric acid. PO_3 .	0.31799	0.63598	0.95397	1.27196	1.58995	1.90794	2.22593	2.54392	2.86191
Phosphate of lime. $2 \text{Ca O} + \text{PO}_3$.	Phosphoric acid. PO_3 .	0.55618	1.11236	1.66854	2.22472	2.78090	3.33708	3.89326	4.44944	5.00562
Phosphate of lead. $2 \text{Pb O} + \text{PO}_3$.	Phosphoric acid. PO_3 .	0.24239	0.48478	0.72717	0.96956	1.21195	1.45434	1.69673	1.93912	2.18151
Platinum. Pt .	Ammonia. NH_3 .	0.17390	0.34780	0.52170	0.69560	0.86950	1.04340	1.21730	1.48120	1.56510
Platinum, chloride of potassium and. $\text{Pt Cl}_2 + \text{K Cl}$.	Platinum. Pt .	0.40420	0.80840	1.21260	1.61680	2.02100	2.42520	2.82940	3.23360	3.63780
Platinum, chloride of ammonium and. $\text{Pt Cl}_2 + \text{NH}_4 \text{ Cl}$.	Platinum. Pt .	0.44232	0.88464	1.32696	1.76928	2.21160	3.65392	3.09624	3.53856	3.98088
Potash. KO .	Potassium. K .	0.83048	1.66097	2.49145	3.32194	4.15242	4.98290	5.81339	6.64387	7.47436

Found.	Required.	1.	2.	3.	4.	5.	6.	7.	8.	9.
Potash, sulphate of. KO, SO ₅ .	Potash. KO.	0.54067	1.08134	1.62201	2.16268	2.70335	3.24402	3.78469	4.32536	4.86603
Potash, carbonate of. KO, CO ₂ .	Potash. KO.	0.68092	1.36184	2.04276	2.72368	3.40460	4.08552	4.76644	5.44736	6.12828
Potassium, chloride of. K Cl.	Potassium. K.	0.52534	1.05068	1.57602	2.10136	2.62670	3.15204	3.67738	4.20272	4.72806
Potassium, chloride of platinum and. Pt Cl ₂ + K Cl.	Potash. KO.	0.19334	0.38668	0.58002	0.77366	0.96670	1.16004	1.35338	1.54672	1.74006
Selenious acid. Se O ₂ .	Selenium. Se.	0.71206	1.42411	2.13617	2.84823	3.56028	4.27234	4.98440	5.69646	6.40851
Selenic acid. Se O ₃ .	Selenium. Se.	0.62244	1.24489	1.86733	2.48977	3.11221	3.73466	4.35710	4.97954	5.60199
Selenium, sulphuret of. Se S ₂ .	Selenium. S.	0.55143	1.10286	1.65429	2.20572	2.75715	3.30858	3.86001	4.41144	4.96287
Selenium, sulphuret of. Se S ₃ .	Selenious acid. Se O ₃ .	0.77442	1.54884	2.32326	3.09768	3.87210	4.64652	5.42094	6.19536	6.96978
Silver, oxide of. Ag O.	Silver. Ag.	0.93111	1.86222	2.79333	3.72444	4.65555	5.58667	6.51778	7.44889	8.38000

Found.	Required.	1.	2.	3.	4.	5.	6.	7.	8.	9.
Silver, chloride of. Ag Cl.	Oxide of silver. Ag O.	0·80903	1·61806	2·42709	3·23612	4·04515	4·85418	5·66321	6·47224	7·28127
Silver, chloride of. Ag Cl.	Silver. Ag.	0·75330	1·50660	2·25990	3·01320	3·76650	4·51980	5·27310	6·02640	6·77970
Soda. Na O.	Sodium. Na.	0·74418	1·48836	2·23253	2·97671	3·72089	4·46507	5·20925	5·95342	6·69760
Soda, sulphate of. Na O, SO ₃ .	Soda. Na O.	0·43819	0·87638	1·31457	1·75276	2·19095	2·62914	3·06733	3·50552	3·94371
Sodium, chloride of. Na Cl.	Soda. Na O.	0·53289	1·06578	1·59867	2·13156	2·66445	3·19734	3·73023	4·26312	4·79601
Sodium, chloride of. Na Cl.	Sodium. Na.	0·39656	0·79312	1·18968	1·58624	1·98280	2·37936	2·77592	3·17248	3·56904
Sulphate of barytes. Ba O, SO ₃ .	Sulphur. S.	0·13797	0·27594	0·41391	0·55188	0·68985	0·82782	0·96579	1·10376	1·24173
Sulphate of barytes. Ba O, SO ₃ .	Sulphurous acid. SO ₂ .	0·27514	0·55028	0·82542	1·10056	1·37570	1·65084	1·92598	2·20112	2·47626
Sulphate of barytes. Ba O, SO ₃ .	Sulphuric acid. SO ₃ .	0·34372	0·68744	1·03116	1·37488	1·71860	2·06232	2·40604	2·74976	3·09348

Found.	Required.	1.	2.	3.	4.	5.	6.	7.	8.	9.
Sulphate of lime. Ca O, SO ₃ .	Sulphuric acid. SO ₃ .	0.58468	1.16936	1.75404	2.33872	2.92340	3.50808	4.09276	4.67744	5.26212
Sulphate of lead. Pb O, SO ₃ .	Sulphur. S.	0.10612	0.21224	0.31836	0.42448	0.53060	0.63672	0.74284	0.84896	0.95598
Sulphate of lead. Pb O, SO ₃ .	Sulphuric acid. SO ₃ .	0.26437	0.52874	0.79311	1.05748	1.32185	1.58622	1.85059	2.11496	2.37933
Sulphuret of copper. Cu S.	Sulphur. S.	0.33704	0.67408	1.01112	1.34816	1.68520	2.02224	2.35928	2.69632	3.03336
Sulphuret of silver. Ag S.	Sulphur. S.	0.12955	0.25910	0.38865	0.51820	0.64775	0.77730	0.90685	1.03640	1.16595
Sulphuret of mercury. Hg S.	Sulphur. S.	0.13713	0.27426	0.41139	0.54852	0.68565	0.82278	0.95991	1.09704	1.23417
Strontian. Sr O.	Strontium. Sr.	0.84551	1.69102	2.53653	3.38204	4.22755	5.07306	5.91857	6.76408	7.60959
Strontian, sul- phate of. Sr O, SO ₃ .	Strontian. Sr O.	0.56360	1.12720	1.69080	2.25440	2.81800	3.38160	3.94520	4.50880	5.07240
Strontian, car- bonate of. Sr O, CO ₂ .	Strontian. Sr O.	0.70074	1.40148	2.10222	2.80296	3.50370	4.20444	4.90518	5.60592	6.30666

Found.	Required.	1.	2.	3.	4.	5.	6.	7.	8.	9.
Tin, peroxide of. Sn O ₂ .	Tin. Sn.	0·78616	1·57233	2·35849	3·14466	3·93082	4·71698	5·50315	6·28931	7·07547
Tin, peroxide of. Sn O ₂ .	Protoxide of tin. Sn O.	0·89308	1·78616	2·67924	3·57232	4·46540	5·35848	6·25156	7·14464	8·03772
Titanic acid. Ti O ₂ .	Titanium. Ti.	0·60293	1·20585	1·80878	2·41171	3·01463	3·61756	4·22049	4·82342	5·42634
Titanium, sulphuret of. Ti S ₂ .	Titanic acid. Ti O ₂ .	0·71342	1·42684	2·14026	2·85368	3·56710	4·28052	4·99394	5·70736	6·42078
Tungstic acid. Tu O ₃ .	Tungsten. Tu.	0·79773	1·59547	2·39320	3·19094	3·98867	4·78641	5·58414	6·38188	7·17961
Uranium, peroxide of. U ₂ O ₃ .	Uranium. U ₂ .	0·94758	1·89515	2·84273	3·79031	4·73788	5·68546	6·63304	7·58062	8·52819
Uranium, peroxide of. U ₂ O ₃ .	Protoxide of uranium. 2 UO.	0·98252	1·96504	2·94756	3·93008	4·91260	5·89512	6·87764	7·86016	8·84268
Water HO.	Hydrogen. H.	0·11111	0·22222	0·33334	0·44445	0·55556	0·66667	0·77778	0·88890	1·00001
Water. HO.	Oxygen. O.	0·88889	1·77778	2·66666	3·55555	4·44444	5·33333	6·22222	7·11110	7·99999
Zinc, oxide of. Zn O.	Zinc. Zn.	0·80128	1·60256	2·40385	3·20513	4·00641	4·80769	5·60897	6·41026	7·21154

TABLE OF ATOMIC WEIGHTS, WITH THEIR LOGARITHMS FOR CALCULATIONS IN ANALYSIS.

Name.	Symbol.	Atomic Weight.	Logarithm.
Aluminum - -	Al	171.17	2.233427
Ammonia - -	N H ₃	213.13	2.32864
Antimony - -	Sb	1612.90	3.20761
oxide of - -	Sb O ₃	1912.90	3.28169
sulphuret of - -	Sb S ₃	2216.40	3.34565
Arsenic - -	As	940.10	2.973174
sulphuret of - -	As S ₃	1543.6	3.18854
sulphuret of - -	As S ₅	1945.9	3.28912
Arsenic acid - -	As O ₅	1440.1	3.15839
Arsenious acid - -	As O ₃	1240.1	3.09346
Barium - -	Ba	856.88	2.93292
chloride of - -	Ba Cl	1299.5	3.11378
Barytes - -	Ba O	956.88	2.98086
carbonate of - -	Ba O, CO ₂	1231.9	3.09058
phosphate of - -	2 Ba O, PO ₅	2806.1	3.44811
sulphate of - -	Ba O, SO ₃	1458.1	3.16379
Bismuth - -	Bi	886.92	2.947884
Boron - -	B	136.25	2.134336
Bromine - -	Br	978.31	2.990476
Cadmium - -	Cd	696.77	2.843089
Calcium - -	Ca	256.02	2.408273
chloride of - -	Ca Cl	698.67	2.84427
Carbon - -	C	75.00	1.87506
Carbonic acid - -	CO ₂	275.00	2.43933
Carbonic oxide - -	CO	175.00	2.24304
Cerium - -	Ce	574.70	2.759441
Chlorine - -	Cl	442.65	2.646060
Chromium - -	Cr	351.82	2.546320
Cobalt - -	Co	368.99	2.567015
Columbium (tantalum) - -	Ta	2307.43	3.363128
Copper - -	Cu	395.70	2.59737
protoxide of - -	Cu O	495.70	2.69522
suboxide of - -	Cu ₂ O	891.39	2.95007
Fluorine - -	F	233.8	2.368845
Glucinum - -	G	331.26	2.520168
Gold - -	Au	2486.03	3.395506
Hydrogen - -	H	12.48	1.09621
Iodine - -	I	1579.50	3.198520
Iridium - -	Ir	1233.50	3.091138
Iron - -	Fe	339.21	2.53047
peroxide of - -	Fe ₂ O ₃	978.43	2.99053
protoxide of - -	Fe O	439.21	2.64267
Lead - -	Pb	1294.5	3.11210
carbonate of - -	Pb O, CO ₂	1669.5	3.22259
chloride of - -	Pb Cl	1737.1	3.23983
phosphate of - -	2 Pb O, PO ₅	3681.3	3.56600
protoxide of - -	Pb O	1394.5	3.14442
sulphate of - -	Pb O, SO ₃	1895.7	3.27777

TABLE OF ATOMIC WEIGHTS, WITH THEIR LOGARITHMS FOR CALCULATIONS IN ANALYSIS—*continued.*

Name.	Symbol.	Atomic Weight.	Logarithm.
Lime - - -	Ca O	356.02	2.55147
carbonate of - -	Ca O, CO ₂	631.03	2.80004
sulphate of - -	Ca O, SO ₃	857.19	2.93308
Lithium - - -	L	80.33	1.904878
Magnesia - - -	Mg O	258.35	2.41220
sulphate of - -	Mg O, SO ₃	759.52	2.88054
Magnesium - -	Mg	158.35	2.199618
Manganese - -	Mn	345.9	2.53895
protoxide of - -	Mn O	445.9	2.64924
deutoxide of - -	Mn ₂ O ₃	991.8	2.99642
Mercury - - -	Hg	1265.8	3.10236
subchloride of -	$\frac{1}{2}$ (Hg ₂ Cl)	1487.1	3.17234
oxide of - - -	Hg O	1365.8	3.13539
suboxide of - -	Hg ₂ O	2631.6	3.42022
Molybdenum - -	Mo	598.52	2.777078
Nickel - - -	Ni	369.68	2.567826
Nitrogen - - -	N	175.6	2.24452
Osmium - - -	Os	1244.49	3.094989
Oxygen - - -	O	100.00	2.000000
Palladium - - -	Pd	665.9	2.823409
Phosphoric acid -	Po ₅	892.31	2.95052
Phosphorous acid -	Po ₃	692.21	2.84030
Phosphorus - -	P	392.31	2.59363
Platinum - - -	Pt	1233.3	3.09107
ammonio-chloride of	{ (Pt Cl ₂ + NH ₃ Cl)	2786.5	3.445059
potassio-chloride of	{ (Pt Cl ₂ + K Cl)	3051.2	3.48447
Potash - - -	KO	589.92	2.77079
carbonate of - -	KO, CO ₂	864.93	2.93698
sulphate of - -	KO, SO ₃	1091.1	3.03786
Potassium - - -	K	489.92	2.69013
chloride of - -	K Cl	932.57	2.96968
Rhodium - - -	R	651.39	2.813841
Selenium - - -	Se	494.58	2.694236
Silicon - - -	Si	277.31	2.442965
Silver - - -	Ag	1351.6	3.13085
chloride of - -	Ag Cl	1794.3	3.25389
oxide of - - -	Ag O	1451.6	3.16185
ditto - - -	2	2903.2	3.46288
ditto - - -	3	4354.8	3.63897
ditto - - -	4	5806.4	3.76391
ditto - - -	5	7258.0	3.86082
Soda - - -	Na O	390.9	2.59207
carbonate of - -	Na O, CO ₂	665.91	2.82341
sulphate of - -	Na O, SO ₃	892.07	2.95040
Sodium - - -	Na	290.9	2.46374
chloride of - -	Na Cl	733.55	2.86543

TABLE OF ATOMIC WEIGHTS, WITH THEIR LOGARITHMS FOR CALCULATIONS IN ANALYSIS—*continued*.

Name.	Symbol.	Atomic Weight.	Logarithm.
Strontian - -	Sr O	647·29	2·81110
carbonate of - -	Sr O, CO ₂	922·3	2·96487
sulphate of - -	Sr O, SO ₃	1148·5	3·060131
Strontium - -	Sr	547·29	2·738217
chloride of - -	Sr Cl	989·94	2·99561
Sulphur - -	S	201·17	2·30356
Sulphuric acid - -	S O ₃	501·17	2·69998
Sulphurous acid - -	S O ₂	401·17	2·60333
Tellurium - -	Te	801·76	2·904044
Thorium - -	Th	744·9	2·872098
Tin - -	Sn	735·29	2·86645
peroxide of - -	Sn O ₂	935·29	2·97095
protoxide of - -	Sn O	835·29	2·92184
Titanium - -	Ti	303·66	2·482387
Tungsten - -	W	1183·00	3·072985
Uranium - -	U	2711·36	3·433187
Vanadium - -	V	856·89	2·932924
Water - -	HO,	112·48	2·05107
Ditto - -	2	224·96	2·35210
Ditto - -	3	337·44	2·52820
Ditto - -	4	449·92	2·65314
Ditto - -	5	562·40	2·75005
Ditto - -	6	674·88	2·82923
Ditto - -	7	787·36	2·89618
Ditto - -	8	899·84	2·95417
Ditto - -	9	1012·3	3·00531
Ditto - -	10	1124·8	3·05107
Ditto - -	11	1237·3	3·09248
Ditto - -	12	1349·8	3·13027
Ditto - -	13	1462·2	3·16501
Yttrium - -	Y	402·51	2·604776
Zinc - -	Zn	403·23	2·60555
oxide of - -	Zn O	503·23	2·70177
	2	1006·5	3·00281
	3	1509·7	3·17889
	4	2012·9	3·30382
Zirconium - -	Zr	420·20	2·623456

INDEX.

ABBREVIATIONS, list of, 16.

- Acid, acetic, behaviour of, with reagents, 46, 47.
- , antimonie, behaviour of, with reagents, 42—45.
- , antimonie, estimation of, 199.
- , antimonious, behaviour of, with reagents, 42—45.
- , arsenic, behaviour of, with reagents, 42—45.
- , arsenic, estimation of, 202.
- , arsenious, behaviour of, with reagents, 42—45.
- , boracic, behaviour of, with reagents, 46, 47.
- , bromic, behaviour of, with reagents, 46, 47.
- , carbonic, behaviour of, with reagents, 46, 47.
- , chloric, behaviour of, with reagents, 46, 47.
- , chromic, behaviour of, with reagents, 42—45.
- , chromic, estimation of, 170.
- , citric, behaviour of, with reagents, 46, 47.
- , formic, behaviour of, with reagents, 46, 47.
- , hydrobromic, behaviour of, with reagents, 48, 49.
- , hydrochloric, 12.
- , hydrochloric, behaviour of, with reagents, 48, 49.
- , hydrochloric, detection of, in organic mixtures, 123.
- , hydrochloric, estimation of, 217.
- , hydrocyanic, behaviour of, with reagents, 48, 49.
- , hydrocyanic, detection of, in organic mixtures, 124.
- , hydrofluoric, behaviour of, with reagents, 48, 49.
- , hydriodic, behaviour of, with reagents, 48, 49.
- , hydroselenic, behaviour of, with reagents, 48, 49.

- Acid, hydrosulphocyanic, behaviour of, with reagents, 48, 49.
- , hydrosulphuric, behaviour of, with reagents, 48, 49.
- , hypermanganic, behaviour of, with reagents, 42—45.
- , hyposulphuric, behaviour of, with reagents, 50, 51.
- , hyposulphurous, behaviour of, with reagents, 50, 51.
- , iodie, behaviour of, with reagents, 50, 51.
- , manganic, behaviour of, with reagents, 42—45.
- , molybdic, behaviour of, before blowpipe, 102, 103.
- , molybdic, behaviour of, with reagents, 42—45.
- , nitric, 13.
- , nitric, behaviour of, with reagents, 50, 51.
- , nitric, detection of, in organic mixtures, 122.
- , nitric, estimation of, 228.
- , nitrous, behaviour of, with reagents, 50, 51.
- , osmic, behaviour of, with reagents, 42—45.
- , oxalic, 13.
- , oxalic, behaviour of, with reagents, 50, 51.
- , oxalic, detection of, in organic mixtures, 123.
- , perchloric, behaviour of, with reagents, 50, 51.
- , phosphoric, behaviour of, with reagents, 52, 53.
- , phosphorous, behaviour of, with reagents, 52, 53.
- , selenic, behaviour of, with reagents, 52, 53.
- , selenious, behaviour of, with reagents, 42—45.
- , silicic, behaviour of, with reagents, 52, 53.
- , silicic, blowpipe test for, 104.
- , silicic, estimation of, 207.

- Acid, sulphuric, behaviour of, with reagents, 52, 53.
 —, sulphuric, detection of, in organic mixtures, 122.
 —, sulphuric, estimation of, 212.
 —, sulphurous, behaviour of, with reagents, 52, 53.
 —, sulphurous, estimation of, 213.
 —, tartaric, 13.
 —, tartaric, behaviour of, with reagents, 52, 53.
 —, titanio, estimation of, 171.
 —, tungstic, behaviour of, with reagents, 42—45.
 —, tungstic, behaviour of, before blowpipe, 102, 103.
 —, vanadic, behaviour of, with reagents, 42—45.
 —, uranic, estimation of, 169.
 Alkalies, behaviour of, before blowpipe, 98, 99.
 —, estimation of, in silicates, 210.
 —, fixed, separation of, from alumina, 146.
 —, separation of, from lime, 139.
 —, separation of, from magnesia, 142.
 Alkaline mineral waters, analysis of, 247.
 — sulphurets, analysis of, 216.
 Alkalimeter, 132.
 Alkalimetry, 132.
 Alumina, behaviour of, before blowpipe, 98, 99.
 —, behaviour of, with reagents, 22—25.
 —, estimation of, 144.
 —, separation of, from barytes, 146.
 —, separation of, from fixed alkalies, 146.
 —, separation of, from glucina, 148.
 —, separation of, from lime, 145.
 —, separation of, from phosphoric acid, 225.
 —, separation of, from protoxide of manganese, 150.
 —, separation of, from magnesia, 145.
 —, separation of, from magnesia, lime and alkalies, 144.
 —, separation of, from peroxide of iron, 155.
 —, separation of, from strontian, 146.
 Aluminous minerals, solution of, 146.
 Amalgams, estimation of, mercury in, 185.
 Ammonia, 14.
 —, behaviour of, with reagents, 18—21.
 —, carbonate of, 14.
 —, estimation of, 135.
 Ammonia, hydrosulphate of, 15.
 —, muriate of, 15.
 —, oxalate of, 13.
 Ammonio-nitrate of silver, 118.
 Ammonio-sulphate of copper, 118.
 Alloys, estimation of silver in, 179.
 Analysis of alkaline mineral waters, 247.
 — of alkaline sulphurets, 216.
 — of borate of barytes, 228.
 — of borates, 227.
 — of brass, 178.
 — of bromides, 222.
 — of calomel, 218.
 — of carbonated waters, 239.
 — of carbonates, 233.
 — of cast-iron, 236.
 — of chalybeate waters, 239.
 — of chloride of lead, 217.
 — of chloride of silver, 217.
 — of chlorides, 217.
 — of chromate of lead, 173.
 — of chrome-iron, 170.
 — of clay ironstone, 155.
 — of coal gas, 114.
 — of colophonite, 209.
 — of earthy sulphurets, 216.
 — of fluorides, 222.
 — of gunpowder, 235.
 — of iodides, 220.
 — of metallic sulphurets, 214.
 — of molybdate of lead, 200.
 — of native oxide of tin, 196.
 — of natural silicates, 207.
 — of phosphate of barytes, 224.
 — of phosphate of lead, 224.
 — of platinum ore, 186.
 — of platinum ore, table of, 193, 194.
 — of saline mineral waters, 239.
 — of sulphate of barytes, 213.
 — of sulphate of lead, 212.
 — of sulphate of lime, 213.
 — of sulphate of strontian, 213.
 — of sulphureous mineral waters, 248.
 — of sulphuret of bismuth, 215.
 — of sulphuret of lead, 215.
 — of sulphuret of mercury, 215.
 — of sulphuret of silver, 215.
 — of tungstate of lime, 201.
 — of volatile organic substances, 261.
 — of wolfram, 202.
 —, organic, 254.
 —, organic, by chromate of lead, 263.
 —, organic, by mixture of oxide of copper and chlorate of potash, 262.
 —, organic, determination of nitrogen in, 264.

- Analysis, qualitative, 54.
 —, qualitative, of Berlin silver, 76.
 —, qualitative, of hornblende, 84.
 —, qualitative, of lepidolite, 84.
 —, qualitative, of mineral waters, 85.
 —, qualitative, of Newton's fusible metal, 77.
 —, qualitative, of silicates, 78.
 —, qualitative, of stilbite, 84.
 —, qualitative, of urine, 124.
 —, quantitative, of mineral waters, 239.
 —, tables for calculations in, 281—296.
- Antimonic acid, behaviour of, with reagents, 42—45.
 —, estimation of, 199.
- Antimonious acid, behaviour of, with reagents, 42—45.
- Antimoniurets, blowpipe test for, 105.
- Antimony, detection of, in organic mixtures, 120.
 —, estimation of, 196.
 —, oxide of, behaviour of, before blowpipe, 100, 101.
 —, oxide of, behaviour of, with reagents, 30—33.
 —, separation of, from arsenic, 205.
 —, separation of, from other metals, 198.
 —, separation of, from tin, 199.
- Arsenates, blowpipe tests for, 105.
- Arsenic, detection of, in organic mixtures, 115.
 —, estimation of, 202.
 —, reduction test for, 118.
 —, separation of, from antimony, 205.
 —, separation of, from other metals, 204.
- Arsenic acid, behaviour of, with reagents, 42—45.
 —, separation of, from oxide of zinc, note, 204.
 —, separation of, from other bodies, 204.
- Arsenious acid, behaviour of, with reagents, 42—45.
 —, separation of, from other bodies, 204.
- Arsenites, blowpipe test for, 105.
- Atomic constitution, how to calculate, 269.
- Atomic weights, with their logarithms, 297—299.
- Barium, chloride of, 14.
 —, estimation of, 136.
- Barytes, acetate of, 15.
 Barytes, analysis of borate of, 228.
 —, analysis of sulphate of, 213.
- Barytes, behaviour of, before blowpipe, 98, 99.
 —, behaviour of, with reagents, 18—21.
 —, estimation of, 136.
 —, nitrate of, 14.
 —, separation of, from alumina, 146.
 —, separation of, from lime, 139.
 —, separation of, from strontian, 137.
- Berlin silver, qualitative analysis of, 76.
- Bismuth, analysis of sulphuret of, 215.
 —, estimation of, 174.
 —, oxide of, behaviour of, before blowpipe, 100, 101.
 —, oxide of, behaviour of, with reagents, 30—33.
 —, separation of, from lead, 175.
- Black's blowpipe, 93.
- Blowpipe, use of, in qualitative analysis, 92.
- Boracic acid, behaviour of, with reagents, 46, 47.
 —, estimation of, 227.
- Borate of barytes, analysis of, 228.
- Borates, analysis of, 227.
 —, blowpipe tests for, 104.
- Borax, colours of beads of, 109, 110.
- Brass, analysis of, 178.
- Bromic acid, behaviour of, with reagents, 46, 47.
- Bromides, analysis of, 222.
 —, blowpipe test for, 105.
- Bromine, estimation of, 222.
 —, separation of, from iodine, 222.
- Cadmium, estimation of, 174.
 —, oxide of, behaviour of, before blowpipe, 100, 101.
 —, oxide of, behaviour of, with reagents, 30—33.
 —, estimation of, 138.
- Calculations, tables for, in analysis, 281—296.
- Calculi containing silica, 127.
- Calculus, ammonio-magnesian phosphate, 126.
 —, bone-earth, 126.
 —, carbonate of lime, 127.
 —, cystic oxide, 126.
 —, fusible, 126.
 —, oxalate of lime, 127.
 —, uric acid, 126.
 —, xanthic oxide, 127.
- Calomel, analysis of, 218.
- Carbonated mineral waters, analysis of, 239.
- Carbonates, analysis of, 233.
- Carbon, estimation of, in organic bodies, 254.
 —, separation of, from iron, 236.

- Carbon, separation of, from sulphur, 235.
- Carbonic acid, behaviour of, with reagents, 46, 47.
- Carbonic acid gas, how to determine quantity of, in atmosphere, 232.
- Cast-iron, analysis of, 236.
- Cerium, estimation of, 149.
- , oxide of, behaviour of, before blowpipe, 100, 101.
- , oxide of, behaviour of, with reagents, 22—25.
- Chalybeate mineral waters, analysis of, 239.
- Chloric acid, behaviour of, with reagents, 46, 47.
- Chlorides, analysis of, 217.
- , blowpipe test for, 105.
- Chlorimetry, 218.; Appendix, 279.
- Chlorine, estimation of, 217.
- , separation of, from iodine, 221.
- Chromate of lead, analysis of, 173.
- , use of, in organic analysis, 263.
- Chromic acid, behaviour of, with reagents, 42—45.
- Chrome-iron, analysis of, 170.
- Chromic acid, estimation of, 170.
- Chromium, estimation of, 170.
- , oxide of, behaviour of, before blowpipe, 100, 101.
- , oxide of, behaviour of, with reagents, 22—25.
- , separation of oxide of, from oxide of iron, 170.
- Citric acid, behaviour of, with reagents, 46, 47.
- Clay ironstone, analysis of, 155.
- Coal gas, analysis of, 114.
- Cobalt, estimation of, 159.
- , oxide of, behaviour of, before blowpipe, 100, 101.
- , oxide of, behaviour of, with reagents, 22—25.
- , separation of oxide of, from oxide of nickel, 161.
- , separation of oxide of, from oxide of zinc, 165.
- , separation of oxide of, from peroxide of iron, 164.
- Colophonite, analysis of, 209.
- Constitution, how to calculate atomic, 269.
- Copper, detection of, in organic mixtures, 120.
- , estimation of, 176.
- , oxide of, behaviour of, before blowpipe, 100, 101.
- , protoxide of, behaviour of, with reagents, 30—33.
- Copper, estimation of suboxide of, 178.
- , preparation of oxide of, for organic analysis, 256.
- , separation of, from lead, 178.
- , separation of oxide of, from protoxide of mercury, 185.
- , separation of, from zinc, 178.
- , suboxide of, behaviour of, with reagents, 30—33.
- Cupellation, 180.
- Density, 270.
- of gases, how to determine, 274.
- of liquids, how to determine, 271.
- of solids, how to determine, 272.
- of vapours, how to determine, 277.
- Earths, behaviour of, before blowpipe, 98, 99.
- Earthy sulphurets, analysis of, 216.
- Endiometer, Dr. Ure's, 231.
- Endimetry, 230.
- Filtration, 6.
- Filter, ignition of, 8.
- Flame, construction of, 94.
- Flame, oxidating, 94.
- , reducing, 95.
- Fluorides, analysis of, 222.
- , blowpipe test for, 105.
- Fluorine, estimation of, 222.
- Formic acid, behaviour of, with reagents, 46, 47.
- Fusible metal, qualitative analysis of Newton's, 77.
- Gas, coal, analysis of, 114.
- Gases, change in bulk of, by aqueous vapour, 276.
- Gases, corrections for changes in bulk of, note, 275.
- Gases, how to determine density of, 274.
- Gases, mixed, analysis of, 111, et seq.
- Glucina, behaviour of, before blowpipe, 98, 99.
- , behaviour of, with reagents, 22—25.
- , estimation of, 147.
- , separation of, from alumina, 148.
- , separation of, from magnesia, 148.
- Gold, estimation of, 180.
- , oxide of, behaviour of, with reagents, 30—33.
- , separation of, from silver, 181.

- Griffin's blowpipe, 93.
 Gunpowder, analysis of, 235.
- Hardness of waters, determination of degree of, 249.
- Hornblende, qualitative analysis of, 48.
- Hydriodic acid, behaviour of, with reagents, 48, 49.
- Hydrobromic acid, behaviour of, with reagents, 48, 49.
- Hydrochloric acid, behaviour of, with reagents, 48, 49.
 —, detection of, in organic mixtures, 123.
 —, estimation of, 217.
- Hydrocyanic acid, behaviour of, with reagents, 48, 49.
 —, detection of, in organic mixtures, 124.
- Hydrofluoric acid, behaviour of, with reagents, 48, 49.
- Hydrogen, estimation of, in organic bodies, 254.
- Hydroselenic acid, behaviour of, with reagents, 48, 49.
- Hydrosulphocyanic acid, behaviour of, with reagents, 48, 49.
- Hydrosulphuric acid, behaviour of, with reagents, 48, 49.
- Hypermanganic acid, behaviour of, with reagents, 42—45.
- Hyposulphuric acid, behaviour of, with reagents, 50, 51.
- Hyposulphurous acid, behaviour of, with reagents, 50, 51.
- Iodic acid, behaviour of, with reagents, 50, 51.
- Iodides, analysis of, 220.
 —, blowpipe test for, 105.
 —, estimation of, 220.
- Iodine ley, estimation of iodine in, 221.
- Iodine, separation of, from bromine, 222.
 —, separation of, from chlorine, 221.
- Iridic oxide, behaviour of, with reagents, 30—33.
- Iridium, estimation of, 186.
- Iron, analysis of cast, 236.
 —, estimation of, 153.
 —, peroxide of, behaviour of, before blowpipe, 100, 101.
 —, peroxide of, behaviour of, with reagents, 22—25.
 —, protoxide of, behaviour of, with reagents, 22—25.
 —, separation of, from carbon, 236.
 —, separation of, from other bodies, 153.
- Iron, separation of oxide of, from oxide of chromium, 170.
 —, separation of peroxide of, from alumina, 155.
 —, separation of peroxide of, from earths and alkalies, 155.
 —, separation of peroxide of, from magnesia, 154.
 —, separation of peroxide of, from oxide of cobalt, 164.
 —, separation of peroxide of, from oxide of nickel, 157.
 —, separation of peroxide of, from oxide of zinc, 168.
 —, separation of peroxide of, from protoxide of iron, 155., note.
 —, separation of peroxide of, from protoxide of manganese, 154.
 —, separation of uranium from, 169.
- Ironstone, clay, analysis of, 155.
- Lamp, Berzelius's, for blowpipe experiments, 95.
 —, Rose's, 9.
- Lanthanum, oxide of, behaviour of, with reagents, 22—25.
- Lead, acetate of, 15.
 —, analysis of chloride of, 217.
 —, analysis of chromate of, 173.
 —, analysis of molybdate of, 200.
 —, analysis of phosphate of, 224.
 —, analysis of sulphate of, 212.
 —, analysis of sulphuret of, 215.
 —, detection of, in organic mixtures, 121.
 —, estimation of, 172.
 —, oxide of, behaviour of, before blowpipe, 100, 101.
 —, oxide of, behaviour of, with reagents, 34—37.
 —, separation of, from bismuth, 175.
 —, separation of, from copper, 178.
- Lepidolite, qualitative analysis of, 84.
- Lime, analysis of sulphate of, 213.
 —, analysis of tungstate of, 201.
 —, behaviour of, before blowpipe, 98, 99.
 —, behaviour of, with reagents, 18—21.
 —, estimation of, 138.
 —, separation of, from alkalies, 139.
 —, separation of, from alumina, 145.
 —, separation of, from barytes, 139.
 —, separation of, from magnesia, 141.
 —, separation of, from protoxide of manganese, 156.
 —, separation of, from strontian, 138.

- Liquids, how to determine density of, 271.
- Lithia, behaviour of, before blowpipe, 98, 99.
- , behaviour of, with reagents, 18—21.
- , estimation of, 135.
- , separation of, from potash and soda, 136.
- Logarithms of atomic weights, 297—299.
- Magnesia, behaviour of, before blowpipe, 98, 99.
- , behaviour of, with reagents, 18—21.
- , Magnesia, estimation of, 139.
- , separation of, from alumina, 145.
- , separation of, from fixed alkalies, 142.
- , separation of, from glucina, 148.
- , separation of, from lime, 141.
- , separation of, from peroxide of iron, 154.
- , separation of, from protoxide of manganese, 151.
- Manganese, dentoxide of, behaviour of, with reagents, 22—25.
- , estimation of, 150.
- , protoxide of, behaviour of, with reagents, 22—25.
- , oxide of, behaviour of, before blowpipe, 100, 101.
- , separation of protoxide of, from alumina, 150.
- , separation of, from nickel, 158.
- , separation of protoxide of, from magnesia and lime, 151.
- , separation of protoxide of, from oxide of zinc, 165.
- , separation of protoxide of, from peroxide of iron, 154.
- , valuation of peroxide of, 152.
- Marsh's test for arsenic, 116.
- Mercury, analysis of subchloride of, 218.
- , analysis of sulphurets of, 215.
- , detection of, in organic mixtures, 120.
- , estimation of, 182.
- , estimation of, in amalgams, 185.
- , protoxide of, behaviour of, with reagents, 34—37.
- , separation of protoxide of, from oxide of copper, 185.
- , separation of oxides of, from oxide of silver, 184.
- , suboxide of, behaviour of, with reagents, 34—37.
- Metallic oxides, classification of, 17.
- Microcosmic salt, colours of beads of, 110, 111.
- Mineral waters, analysis of alkaline, 247.
- , qualitative analysis of 85.
- , quantitative analysis of, 239.
- , analysis of sulphureous, 248.
- Molybdate of lead, analysis of, 200.
- Molybdeum, estimation of, 200.
- Molybdic acid, behaviour of, before blowpipe, 102, 103.
- acid, behaviour of, with reagents, 42—45.
- acid, estimation of, 200.
- oxide, behaviour of, with reagents, 34—37.
- Molybdous oxide, behaviour of, with reagents, 34—37.
- Nickel, oxide of, behaviour of, before blowpipe, 102, 103.
- , oxide of, behaviour of, with reagents, 26—29.
- , estimation of, 156.
- , separation of alumina from oxide of, 159.
- , separation of, from manganese, 158.
- , separation of magnesia from oxide of, 159.
- , separation of oxide of, from oxide of cobalt, 161.
- , separation of oxide of, from oxide of zinc, 165.
- , separation of oxide of, from peroxide of iron, 157.
- Nitrates, blowpipe test for, 104.
- Nitric acid, behaviour of, with reagents, 50, 51.
- , detection of, in organic mixtures, 122.
- , estimation of, 228.
- Nitrogen, determination of, in organic substances, 264.
- Nitrous acid, behaviour of, with reagents, 50, 51.
- Organic analysis, 254.
- Organic substances, analysis of volatile, 261.
- Osmic oxide, behaviour of, with reagents, 34—37.
- Osmic acid, behaviour of, with reagents, 42—45.
- Osmium, estimation of, 186.
- Oxalic acid, behaviour of, with reagents, 50, 51.

- Oxalic acid, detection of, in organic mixtures, 123.
- Oxides, metallic, behaviour of, before blowpipe, 100—103.
- Palladious oxide, behaviour of, with reagents, 34—37.
- Palladium, estimation of, 186.
- Perchloric acid, behaviour of, with reagents, 50, 51.
- Platinic oxide, 38—41.
- Platinous oxide, behaviour of, with reagents, 38—41.
- Platinum, estimation of, 186.
- Platinum ore, analysis of, 186.
- , tables of the analysis of, 193, 194.
- Phosphate of barytes, analysis of, 224.
- Phosphate of lead, analysis of, 224.
- Phosphates, blowpipe test for, 104.
- Phosphoric acid, behaviour of, with reagents, 52, 53.
- , estimation of, 224.
- , separation of, from alumina, 225.
- , separation of, from metallic oxides, 225.
- , separation of, from sulphuric acid, 225.
- Phosphorous acid, behaviour of, with reagents, 52, 53.
- Poisons, detection of, in organic mixtures, 115.
- Potash, behaviour of, before blowpipe, 98, 99.
- , behaviour of, with reagents, 18—21.
- , bicarbonate of, 14.
- , binoxalate of, 13.
- , carbonate of, 14.
- , caustic, 13.
- , chromate of, 15.
- , estimation of, 130.
- , separation of, from soda, 131.
- Potassium, estimation of, 130.
- , iodide of, 16.
- Precipitation, 4—6.
- Qualitative analysis, manipulations in, 1.
- Quantitative analysis, manipulations in, 4.
- , observations on, 128.
- Reagent, 2.
- Reagents, list of, with impurities, 12.
- Reduction, test for arsenic, 118.
- Rhodic oxide, behaviour of, with reagents, 38—41.
- Rhodium, estimation of, 186.
- Saline mineral waters, analysis of, 239.
- Seleniates, blowpipe test for, 104.
- Selenic acid, behaviour of, with reagents, 52, 53.
- Selenious acid, behaviour of, with reagents, 42—45.
- Selenites, blowpipe test for, 104.
- Seleniurets, blowpipe tests for, 105.
- Silica, estimation of, 206.
- Silicates, analysis of natural, 78, 207.
- , constituents of, 80.
- , estimation of alkalies in, 210.
- , list of, decomposable by hydrochloric acid, 208.
- , list of, not decomposable by hydrochloric acid, 208.
- , qualitative analysis of, 78.
- , solution of, 78.
- Silicic acid, behaviour of, with reagents, 52, 53.
- , blowpipe test for, 104.
- , estimation of, 207.
- Silver, analysis of chloride of, 217.
- , analysis of sulphuret of, 215.
- , estimation of, 179.
- , nitrate of, 14.
- , oxide of, behaviour of, before blowpipe, 102, 103.
- , oxide of, behaviour of, with reagents, 38—41.
- , separation of, from gold, 181.
- , separation of oxide of, from oxides of mercury, 184.
- Soda, behaviour of, before blowpipe, 98, 99.
- , behaviour of, with reagents, 18—21.
- , carbonate of, 14.
- , estimation of, 131.
- , separation of, from potash, 131.
- , sulphate of, 12.
- Sodium, estimation of, 131.
- Solids, how to determine density of, 272.
- Solution of aluminous minerals, 146.
- Solution of silicates, 78.
- Specific gravity, 270.
- gravity of gases, how to determine, 274.
- gravity of liquids, how to determine, 271.
- gravity of solids, how to determine, 272.
- gravity of vapours, how to determine, 277.
- Stilbite, qualitative analysis of, 84.
- Strontian, analysis of sulphate of, 213.
- , behaviour of, before blowpipe, 98, 99.

- Strontian, behaviour of, with reagents, 18—21.
 —, estimation of, 137.
 —, separation of, from alumina, 146.
 —, separation of, from barytes, 137.
 —, separation of, from lime, 138.
 Strontium, estimation of, 137.
 Sulphate of barytes, analysis of, 213.
 — of lead, analysis of, 212.
 — of lime, analysis of, 213.
 — of strontian, analysis of, 203.
 Sulphates, blowpipe tests for, 104.
 Sulphureous mineral waters, analysis of, 248.
 Sulphuret of bismuth, analysis of, 215.
 — of lead, analysis of, 215.
 — of mercury, analysis of, 215.
 — of silver, analysis of, 215.
 Sulphuretted hydrogen, 14.
 Sulphurets, analysis of alkaline, 216.
 —, analysis of earthy, 216.
 —, analysis of metallic, 214.
 —, blowpipe tests for, 105.
 Sulphuric acid, 12.
 —, behaviour of, with reagents, 52, 53.
 —, detection of, in organic mixtures, 122.
 —, estimation of, 212.
 Sulphurous acid, behaviour of, with reagents, 52, 53.
 —, estimation of, 213.
 Sulphur, separation of, from carbon, 235.
 Sulphuric acid, separation of, from phosphoric acid, 225.
 Supports in blowpipe experiments, 95.
 Symbols, list of, 16.
 Tantalalic acid, behaviour of, with reagents, 26—29.
 Tantalum, oxide of, behaviour of, before blowpipe, 102, 103.
 Tartaric acid, behaviour of, with reagents, 52, 53.
 Telluric oxide, behaviour of, with reagents, 38—41.
 Tellurets, blowpipe test for, 105.
 Tellurium, oxide of, behaviour of, before blowpipe, 102, 103.
 Test glass, 2.
 — tube, 3.
 Thorina, behaviour of, with reagents, 26—29.
 —, estimation of, 147.
 Tin, analysis of, native oxide of, 196.
 —, estimation of, 195.
 —, means of separating other bodies from, 195.
 Tin, oxide of, behaviour of, before blowpipe, 102, 103.
 —, peroxide of, behaviour of, with reagents, 38—41.
 —, protochloride of, 15.
 —, protoxide of, behaviour of, with reagents, 38, 39, 40, 41.
 —, separation from antimony, 199.
 Titanic acid, behaviour of, with reagents, 26, 27, 28, 29.
 —, estimation of, 171.
 Titanium, oxide of, behaviour of, before blowpipe, 102, 103.
 Tungstate of lime, analysis of, 201.
 Tungstic acid, behaviour of, before blowpipe, 102, 103.
 —, behaviour of, with reagents, 42—45.
 —, estimation of, 201.
 Uranium, estimation of, 169.
 —, oxide of, behaviour of, before blowpipe, 102, 103.
 —, oxide of, behaviour of, with reagents, 26—29.
 —, peroxide of, 26—29.
 —, separation of iron from, 169.
 Urine, qualitative analysis of, 124.
 Vapours, how to determine density of, 277.
 Valuation of peroxide of manganese, 152.
 Vanadic acid, behaviour of, with reagents, 42—45.
 Vanadium, oxide of, behaviour of, before blowpipe, 102, 103.
 —, oxide of, behaviour of, with reagents, 26—29.
 Voigt's blowpipe, 93.
 Volatile precipitates, drying and ignition of, 11.
 Water, estimation of, 237.
 Waters, how to determine degree of hardness of, 249.
 —, qualitative analysis of mineral, 85.
 —, quantitative analysis of mineral, 239.
 Weights, atomic, with their logarithms, 295—297.
 Wolfram, analysis of, 202.
 Yttria, behaviour of, before blowpipe, 98, 99.
 —, behaviour of, with reagents, 26—29.
 —, estimation of, 147.

Yttria, separation of, from earths and alkalies, 147.

Zinc, oxide of, behaviour of, before blowpipe, 102, 103.

—, oxide of, behaviour of, with reagents, 26—29.

—, estimation of, 164.

—, separation of arsenic acid from oxide of, 204. note.

—, separation of, from copper, 178.

—, separation of oxide of, from peroxide of iron, 168.

Zinc, separation of oxide of, from oxide of cobalt, 165.

—, separation of oxide of, from oxide of manganese, 165.

—, separation of oxide of, from oxide of nickel, 165.

Zirconia, behaviour of, before blowpipe, 98, 99.

—, behaviour of, with reagents, 26—29.

—, estimation of, 149.

THE END.

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